

Prepared for:

Hercules Incorporated
Hercules Plaza
1313 North Market Street
Wilmington, Delaware 19894-0001

REVISION 0
SAMPLING AND ANALYSIS PLAN
QUALITY ASSURANCE PROJECT PLAN
HEALTH AND SAFETY PLAN

TERRY CREEK SITE
BRUNSWICK, GEORGIA

Prepared by:



GEOSYNTEC CONSULTANTS

1100 Lake Hearn Drive, NE, Suite 200
Atlanta, Georgia 30342

Project Number GQ0270-02

August 1997



15 August 1997

Mr. Leo Francendese
Federal On-Scene Coordinator
USEPA - Region IV
Atlanta Federal Center
100 Alabama Street, SW
Atlanta, Georgia 30303

Subject: Sampling and Analysis Plan,
Quality Assurance Project Plan,
and Health and Safety Plan
Terry Creek Site
Brunswick, Georgia

Dear Mr. Francendese:

Please find enclosed three copies of the Sampling and Analysis Plan, Quality Assurance Project Plan, and Health and Safety Plan for the Terry Creek Site. These plans are being forwarded at the request of Mr. Timothy D. Hassett of Hercules. Please contact Mr. Hassett if you have any questions.

Sincerely,

J. F. Beech, Ph.D., P.E.
Principal

Enclosure

Copy to: T. Hassett, Hercules

GQ0270-02/GA971040.DOC

Corporate Office:

621 N.W. 53rd Street • Suite 650
Boca Raton, Florida 33487 • USA
Tel. (561) 995-0900 • Fax (561) 995-0925

Regional Offices:

Atlanta, GA • Boca Raton, FL • Chicago, IL
Columbia, MD • Huntington Beach, CA • San Antonio, TX
Walnut Creek, CA • Paris, France

Laboratories:

Atlanta, GA
Boca Raton, FL
Huntington Beach, CA



RECYCLED AND RECYCLABLE



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1. INTRODUCTION

1.1 Terms of Reference

This Sampling and Analysis Plan (SAP) was prepared by GeoSyntec Consultants (GeoSyntec) on behalf of Hercules Incorporated (Hercules) for the Terry Creek Site located in Brunswick, Georgia. This SAP is being prepared at the request of the United States Environmental Protection Agency (USEPA), Region IV Emergency Response Group, which is considering a removal action if unacceptable levels of toxaphene are found in sediments at the site.

The scope of this SAP was discussed in a conference call held among Hercules, USEPA and GeoSyntec on 18 June 1997. This document addresses the activities that will be conducted to sample and analyze for the presence of toxaphene in soil and sediment. The primary areas that will be investigated include a dredge spoil area, the marsh areas around the dredge spoil area, the berm surrounding the dredge spoil area, Dupree Creek, portions of Terry Creek, and the Outfall Ditch from the Hercules plant.

This SAP was prepared by Mr. Charles A. Spiers, P.G. and it was reviewed by Dr. J.F. Beech, P.E. in accordance with GeoSyntec's peer review policy.

1.2 Site Description and History

Portions of Terry Creek and Dupree Creek initially were dredged by the Army Corps of Engineers (USACE) between 1938 and 1948. No dredging of these areas occurred again until the early 1970s. During the period in which dredging was not occurring, Hercules began to produce toxaphene, a pesticide, in its plant in Brunswick. Until production of this product was discontinued in 1980, some amounts of this material was contained in legal discharge from the plant. This discharge entered into Dupree Creek.

In 1972, with the approval of the USEPA and the US Fish and Wildlife Services and the State of Georgia, the USACE chose an area on the North side of Terry Creek at the confluence of Terry and Dupree Creeks in which to place dredge spoils from the dredging of Terry and Dupree Creeks (i.e., the Dredge Spoil Area). Dredge spoils were

placed in this area from 1972 until dredging was discontinued in the late 1980s. The Dredge Spoil Area and areas adjoining it are the subject of this report.

The primary disposal area used by the USACE at the Terry Creek Site is the Dredge Spoil Area. The dredge spoil in this area contains toxaphene. The Dredge Spoil Area is surrounded by a berm. Initially, a low-level berm was constructed from marsh sediment. Dredge spoil was placed within the berm and allowed to dewater by gravity drainage. The dewatered dredge material was pushed up against the inside of the original, low-level berm to form a higher inner berm. The dewatered dredge material was not pushed in the front of the original low-level to form a higher inner berm.

1.3 Project Background

In 1995, the USEPA Region IV conducted an Expanded Site Inspection (ESI) of the Terry Creek Site. A total of 45 samples were collected during the ESI that included: groundwater samples, surface and subsurface soil samples, surface water samples and sediment samples collected from Dupree and Terry Creeks and the Back River. Samples of soil and sediment collected in the dredge spoil area, as well as the sediments of Dupree Creek and Terry Creek, contained measurable concentrations of toxaphene (Figure 2).

Data about the Terry Creek Site have been gathered as a result of the ESI, previous sampling events conducted by Hercules, the USACE, the Brunswick Initiative and the State of Georgia Environmental Protection Division (GAEPD). The USEPA Region IV and Hercules are considering a removal action of contaminated soil and sediment in and around the dredge spoil area. USEPA is currently reviewing available data. As part of its evaluation, USEPA has requested additional samples of soil and sediment from the Terry Creek Site be collected and analyzed for toxaphene.

Hercules has committed to further sampling. This SAP addresses sample collection, sample locations, number of samples, methods for sampling and analysis, quality assurance and quality control procedures.

2. SAMPLE COLLECTION

2.1 Overview

This section describes the procedures to be used to obtain samples of soil and sediment in and around the Dredge Spoil Area for toxaphene analyses. The toxaphene analyses will be performed by En Chem, Inc. (En Chem) in Madison, Wisconsin. Surface soil or sediment at each proposed location will be sampled at a depth of 0 to 12 inches. Surface soil and sediment samples will be collected using either stainless steel trowels, spoons, push tubes, or hand augers. At five locations inside the berm of the dredge spoil area, a lysimeter will be installed just above the water table. Liquid collected in the lysimeters will be analyzed for toxaphene. Sediment samples will also be obtained from the Hercules Outfall Ditch to assist Hercules in estimating the volume of sediment containing toxaphene. Sample handling, packing, and documentation are discussed in the following sections. Methods for establishing sample locations are found in Section 3.

Access to the sampling locations will be by boat. Because there is a seven foot tidal fluctuation in the creeks and marsh areas, sampling in some of these areas may only be possible during high tide conditions. Sampling in the Dredge Spoil Area can take place on foot without concern for tidal conditions except for initial daily access.

During sediment sampling in the creeks, tidal conditions will be noted and field water quality parameters (i.e., salinity, temperature, dissolved oxygen and specific conductance) will be measured. The field meters used for measuring these parameters will be calibrated and operated in accordance with the manufacturers' recommendations. Other indicator parameters will be measured in the laboratory for select soil and sediment samples. Geotechnical indicator parameters will include soil particle size distribution (ASTM Method D 422) and Atterberg limits (ASTM Method D 4318). These parameters will be measured by GeoSyntec's Geoenvironmental Laboratory in Alpharetta, Georgia. Also, total organic carbon (TOC) (USEPA Method 415.1) will be measured by En Chem. Appropriate quality assurance and quality control procedures for these tests will be followed. Depositional and lithologic characteristics of the soil and sediments will be noted during sampling.

The sampling program for the Terry Creek site is divided into six zones, which are described in Section 2.2. Sampling requirements for duplicate samples, splitting samples, etc. are also discussed in Section 2.2. There is a slightly different rationale for sampling each zone, but the overall intent is to identify the presence of measurable concentrations of toxaphene that may be included in a removal action performed by Hercules. The distribution of toxaphene in the soil and sediment will be evaluated using geostatistical techniques.

2.2 Sediment and Soil Sampling Program

2.2.1 North Marsh Area

The North Marsh Area consists of the west to east portion of Dupree Creek north of the Dredge Spoil Area, marsh deposits north of the creek, and marsh deposits south of the creek (Figure 3). The purpose of sampling in this area is to ascertain whether toxaphene is present in sediments that may have migrated from the weirs at the northern edge of the Dredge Spoil Area.

Four transects will be sampled in the section of Dupree Creek north of the Dredge Spoil Area. The sampling at each transect will consist of a total three samples taken from each bank and the center of the creek. Depending on the softness of the sediments, either a push sampler, hand auger or Ponar sampler will be used. The sampling depth will be 0 to 12 in. An aliquot will be obtained from the three samples along a transect. In addition, a composite sample will be prepared for each transect. The procedures for preparing the aliquots and composite samples are described below. Specific procedures for sampling and mixing are described in USEPA [1996].

Each sample in a transect will be placed in a separate mixing bowl. After thorough mixing of an individual sample, an aliquot will be taken from the mixing bowl and placed in a clean sample jar. The jar will be sealed with a Teflon-lined screw cap. Once all three aliquots are obtained, the remaining portions of the three transect samples will be placed in a clean mixing bowl. A composite sample will be prepared by mixing the combined samples. A composite sample will be placed in a separate sample jar.

The composite sample along with each of the aliquots will be extracted according to EPA Test Methods for Evaluating Solid Wastes SW-846, Method 3550A. The composite sample extract will be analyzed for toxaphene according to SW-846, Method 8081 and using the specific guidance agreed upon by USEPA Region IV (Appendix C of the QAPP). All samples will be extracted and analyzed by En Chem. The decision to analyze the aliquots for a given transect will depend upon the results of analyses for the composite samples for that and adjacent transects.

Eight samples will be collected in the North Marsh Area. Five samples will be obtained at locations evenly distributed in the marsh south of Dupree Creek. Three samples will be obtained in the marsh north of Dupree Creek. The proposed sample locations are shown on Figure 3.

2.2.2 Dupree Creek

Five transects will be sampled in Dupree Creek, west of the Dredge Spoil Area. The sampling and analysis procedures, including compositing, will be the same as that described in Section 2.2.1. The proposed transect locations are shown in Figure 3.

Two additional sediment samples will be obtained from the west bank of Dupree Creek. The locations of these samples are in the vicinity of the boat dock between the Hercules Outfall Ditch and Terry Creek (Figure 3).

2.2.3 Terry Creek

Seven transects will be sampled in Terry Creek (Figure 3). The sampling and analysis procedures, including compositing, will be the same as that described in Section 2.2.1. The proposed transect locations are shown in Figure 3.

2.2.4 Dredge Spoil Area

Further characterization of the Dredge Spoil Area will consist of twelve surface samples. The twelve samples will be located along four transects in the Dredge Spoil

Area (Figure 3). Lysimeters will be installed at five of the sample locations, just above the water table. Water samples from the lysimeters will be analyzed for toxaphene to determine leachability characteristics. The samples will be extracted according to SW-846, Method 3510B, and the extracts will be analyzed by SW-846 Method 8081, using the specific guidance agreed upon by EPA Region IV (Appendix C of the QAPP). The samples will also be analyzed for Total Organic Carbon (TOC) by USEPA-600/4-79-020 Method 415.2, and Total Suspended Solids (TSS) by USEPA-600/4-79-020 Method 160.2. If toxaphene is detected in the lysimeter samples, the data may be used in a contaminant transport model, AT123D, to estimate the potential for transport of toxaphene in ground water. The modeling will be performed by GeoSyntec.

2.2.5 Berm Area

Twelve surface samples will be collected from the berm around the Dredge Spoil Area. Four samples will be collected on the inside (near the crest) of the berm. These samples are to be obtained from the portion of the berm constructed from dewatered dredge spoil. Eight samples will be collected on the outside of the berm. These samples are to be collected from the portion of the berm constructed from marsh sediment. These samples will be used to evaluate whether the berm contains measurable levels of toxaphene.

An additional sample will be collected at the discharge point from each weir. These samples will be used to evaluate whether discharge from the weir has occurred.

Several seep areas around the berm have been observed by the USEPA during low tide. These seeps are probably a result of bank storage in the base of the berm, which occurs during high tide (tide fluctuations average about 7 feet). In order to estimate the amount of bank storage, five samples will be collected with a push tube (0-2 ft) for permeability testing using ASTM Method D-5084. The locations for these samples are not shown on Figure 3, but will be determined during the field sampling event. These permeability tests will be performed by GeoSyntec.

2.2.6 Hercules Outfall Ditch

Shallow sediments in the outfall ditch have been characterized by previous sampling events (Figure 2). It is proposed that four additional samples be collected from 1 to 2 feet below the surface in order to assess the volume of sediments that contain measurable levels of toxaphene. The proposed sample locations are downstream of the weir structure in the ditch (Figure 3).

2.2.7 Other Samples

Samples in addition to those described in Sections 2.2.1 through 2.2.6 will be collected to confirm the validity of the sampling and analytical procedures. The samples to be collected include equipment rinsate blank, blind duplicates, matrix spike samples and matrix spike duplicate samples.

Equipment rinsate blanks are described in Section 8.3 of the Quality Assurance Project Plan. Equipment rinsate blanks will be collected at the outset of the field sampling program and once a week thereafter.

Blind duplicate samples will be collected at a frequency of 1 every 20 samples. Every 20th sample should not be duplicated. Instead a random selection process should be used that results in a frequency of 1 per 20 samples. Blind duplicate samples are not to be identified as blind duplicates. The sample should be labeled using the sample identification procedure described in Section 2.6. The regular sample and duplicate sample identification are to be the same except the duplicate sample is given the next sequential sample number. Blind duplicate samples will be identified only in the field log book.

Samples will be split with the USEPA in accordance with the procedures outlined in USEPA [1996]. At least one blind duplicate will be split.

Matrix spike and matrix spike duplicate samples will be prepared and analyzed by the laboratory. These samples will be selected and prepared by the laboratory from field samples sent to the analytical laboratory.

2.3 Sample Containers, Preservatives, and Holding Times

Sample containers, preservatives, holding times, and the analytical method to be used for toxaphene are discussed in the Quality Assurance Project Plan (QAPP) [GeoSyntec, 1997]. In summary, SW-846 Method 8081 requires that soil and sediment samples to be analyzed for toxaphene have no more than a 14-day holding time to extraction, and be analyzed within 40 days after extraction. Water samples analyzed for toxaphene are to have no more than a seven day holding time to extraction and are to be analyzed within 40 days of extraction. Samples collected during this program will be placed in 500-milliliter (mL) amber jars with Teflon-sealed caps. Table 1 summarizes the number of samples and supporting quality assurance samples to be collected during the field program. All sample containers for samples submitted for laboratory analyses will be provided by En Chem, Inc. (En Chem). Containers will be new, pre-cleaned, or pre-baked as appropriate.

Sample preservation is not required for soil or sediment samples; however, temperature preservation is required.

2.4 Sample Packing, Handling, and Shipping

2.4.1 Sample Packing

The sediment and soil samples obtained in this sampling program will be placed in shipping coolers with enough ice or freezer packs to maintain a temperature of 4°C, and with sufficient bubble pack to prevent breakage during shipping. Custody seals will be applied to sample jars and shipping containers. All samples in a shipping container will be listed on the chain-of-custody form enclosed in the shipping container (Figure 4). Once the samples are securely packaged, the container will be sealed with tape and several custody seals will be placed over the top edge.

2.4.2 Sample Shipping

All samples will be shipped via courier service (e.g., Federal Express, Airborne, etc.). A signed chain-of-custody form will be enclosed in each container. A shipping document for the courier service will be completed for each shipment.

2.5 Sample Documentation

2.5.1 General

Documents recording sampling events will include a daily field activity log, health and safety log, and field measurement logs. The information to be recorded on these logs are discussed in Section 5. Sample information to be included on sample labels, custody seals, and chain-of-custody forms is described below.

2.5.2 Sample Labels

Each sample bottle will be labeled with the following information: date and time of sample collection, sample number, analyte, project and task number, and sampler's initials. Indelible ink will be used to record information on the sample label. The sample identification procedure described in Section 2.6 will be used.

2.5.3 Custody Seals

Custody seals will be used when a sample shipment is picked up by the laboratory or sent to the laboratory by overnight courier. Custody seals ensure that any tampering during transportation will be detected by the receiving laboratory. Signed and dated custody seals will be placed on each sample jar in such a way that is necessary to break the seal to open the jar. Signed and dated custody seals will be attached to the top of the shipping container in such a way that it is necessary to break the seal to open the container.

2.5.4 Chain-of-Custody Forms

Chain-of-custody forms provide the documentation to trace sample possession from the time of sample collection until receipt by the laboratory. One chain-of-custody form will be filled out for each cooler or shipping container. All the samples contained in the cooler or container will be listed on the chain-of-custody form. An example chain-of-custody form is presented in the QAPP. One copy of the completed form will be placed in a plastic bag taped to the inside lid of the shipping container and one copy will be kept with the project files.

2.6 Sample Identification

Samples that are collected in the field will be identified with a unique alphanumeric identification. The identification will follow the following format:

XXXXXX-YYDDD-ZZ

where:

- XXXXXX is up to a five character description of the origin of the sample. The first two characters will describe the type of sample and the last three will describe the sample location. For example, a soil sample taken from the Dredge Spoil Area will have the description of SLDSA, where SL is an abbreviation for soil and DSA is an abbreviation for the location. Sediment samples will be labeled in a similar fashion as soil, except contain a SD prefix.
- YY is the year which the sample was taken.
- DDD is the date on the Julian calendar.
- ZZ is a sequential number which restarts each day.

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For example, if three samples were taken over the course of one day, the following ID's may be used:

- SDOD-97293-01 (a sediment sample taken from the Outfall Ditch on 19 October 1997);
- SDOD-97293-02 (a second sediment sample taken from the Outfall Ditch on 19 October 1997); and
- SLDSA-97293-03 (a soil sample taken from the Dredge Spoil on 19 October 1997).

3. SAMPLE LOCATIONS WITH GLOBAL POSITIONING SYSTEM (GPS)

3.1 Overview

All sampling points will be located using a global positioning system (GPS), which is a satellite-based positioning system operated by the U.S. Department of Defense (DOD). This system provides all-weather, worldwide, 24-hour position and time information. The positioning accuracy will be + 1 meter for horizontal and vertical control.

3.2 Operating Procedures

The GPS equipment consists of a dual receiver system with a fixed base station and a roving unit. It is possible to perform real time surveying where results can be viewed immediately in the field. The roving unit is carried to each sample location. The rover consists of a back-pack unit with a rod with antenna, similar to a surveying rod. When reading a sampling point, the rod is set down over the point and a coordinate is read and stored in the memory of the unit. The sample ID is also entered and stored. This information will also be recorded in a field notebook to provide backup for the unit.

3.3 Calibration Procedure

Daily calibration of the GPS will be performed by always locating the base station at the same location. It is proposed that the base station be located at a marked point on the Hercules boat dock or survey benchmark at the facility.

A fixed reference point located several hundred feet away will be used to locate the rover each day. The coordinate of the reference point will be compared to the previous day's reference coordinates to confirm the rover is providing the consistent readings for each sampling day. These measurements, or any deviations, will be recorded in the field log book.

4. EQUIPMENT DECONTAMINATION

4.1 Sampling Equipment Decontamination

Field sampling equipment will be decontaminated on-site according to the procedures outlined in Appendix B of the USEPA Region 4 SOP/QAM (USEPA, 1996). The general procedures for decontaminating the equipment are listed below.

1. Wash equipment thoroughly with soap and water using a brush or scrub pad to remove any particulate matter or surface film.
2. Rinse equipment thoroughly with tap water.
3. Rinse equipment thoroughly with analyte-free water.
4. Rinse equipment thoroughly with pesticide-grade hexane and allow to air dry.
5. Wrap equipment in one layer of aluminum foil. Seal the foil-wrapped equipment in plastic.

Tap water from any municipal water treatment system, or distilled/deionized water, may be used for initial equipment rinses. The use of an untreated potable water supply is not an acceptable substitute for tap water.

4.2 Disposal of Decontamination Waste Water and Solids

Disposal of wastewater and solids generated during decontamination of sampling equipment will include containerizing this material in plastic buckets with lids. The material in these buckets will be consolidated in a drum. The materials will be handled according to State and Federal guidance.

5. GENERAL FIELD DOCUMENTATION PROCEDURES AND GUIDELINES

5.1 Overview

General field documentation procedures and guidelines to be used in performing investigations are addressed in this section. If deviations from these procedures are necessary, alternative procedures and the reason for their use will be documented in the appropriate field activity log for that task.

5.2 Field Activity Logs

5.2.1 Introduction

A field log book will be maintained to record the details of field investigation activities and field data. This log book will be bound and will have sequentially numbered pages. Entries will be written in indelible ink and will be initialed and dated by the field personnel recording the information. Corrections to log entries will be made by crossing out incorrect entries and initialing and dating the strike-out. The correct entry then will be made. In addition, several types of field activity logs will be maintained: daily field activity logs, site health and safety logs, equipment calibration logs, and field sampling logs.

5.2.2 Daily Field Activity Logs

A daily field activity log will be completed by the field staff each day work is performed at the site. It will be the responsibility of the site field supervisor to ensure that this log is completed. Information to be provided on the log includes, as appropriate:

- project name/number;
- on-site personnel;

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- date and weather conditions;
- personnel leaving or arriving on site;
- time and description of task activities;
- description of any samples collected;
- record of sampling activities;
- unusual events;
- other progress or problems; and
- communication with others.

The daily field activity log will be signed by the individual who prepares it. Daily field activity logs will be submitted on a weekly basis to the GeoSyntec Project Manager. Following review, the logs will be placed in the project file.

5.2.3 Site Health and Safety Log

The Site Health and Safety Officer (SHSO) will be responsible for maintaining the health and safety log(s) for the various field activities. This log will contain instrument calibration data and a record of the time, location, and concentration of contaminants measured during the various field activities. A separate page will be maintained for readings from monitoring equipment used for each task on a daily basis. The log will include the following information:

- signature of individual filling out the log;
- date/time;
- names of personnel in work zone and their affiliation;
- level of personal protection utilized;

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- weather conditions;
- work activity/task;
- monitoring/sampling information: time, location of measurement (e.g., breathing zone, downhole), measurement value and units, and type of instrument/serial number;
- calibration information: date and time of calibration, calibration results, type of instrument, and instrument serial number;
- any other pertinent information with regard to health and safety (i.e., accidents, equipment entering work zone, decontamination of equipment); and
- equipment calibration information including but not limited to the following: date and time of calibration, name of person conducting calibration, type of instrument and serial number, type of calibration conducted, and calibration results.

5.2.4 Field Sampling Logs

In addition to the descriptions of field investigation activities and field data recorded in the field log book, details of sampling information may be provided on field sampling logs. Types of field sampling logs may include:

- water level measurement logs;
- water sampling logs; and
- soil/sediment sampling logs.

Field sampling logs will generally include the following information:

- date and weather;
- personnel;

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- time and description of investigative activities;
- sample medium and type (i.e., grab, composite, duplicate, etc.);
- sample collection technique(s);
- sample containers, analyses and preservatives;
- sample number, location, and depth;
- sampling times;
- tidal conditions;
- pertinent field observations;
- field parameters (salinity, temperature, dissolved oxygen, and specific conductance); and
- names of samplers.

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6. REFERENCES

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TABLES

TABLE 1
SUMMARY OF SOIL AND SEDIMENT
SAMPLING BY AREA¹⁾

TERRY CREEK SITE
BRUNSWICK, GEORGIA

AREA	SHALLOW SAMPLES	COMPOSITE SAMPLES ⁽²⁾	WATER ⁽³⁾ SAMPLES	FIELD DUPLICATES ⁽⁵⁾	EQUIPMENT BLANKS	TOTAL
North Marsh Area	20	4		2	2	28
Dupree Creek Channel	17	5		2	2	26
Terry Creek Channel	21	7		2	2	32
Dredge Spoil Area	12		5	2	2	21
Dredge Spoil Berm Area ⁽⁴⁾	15			1	1	17
Hercules Outfall Ditch ⁽⁶⁾	4			1	1	6
TOTALS	89	16	5	10	10	130

NOTES:

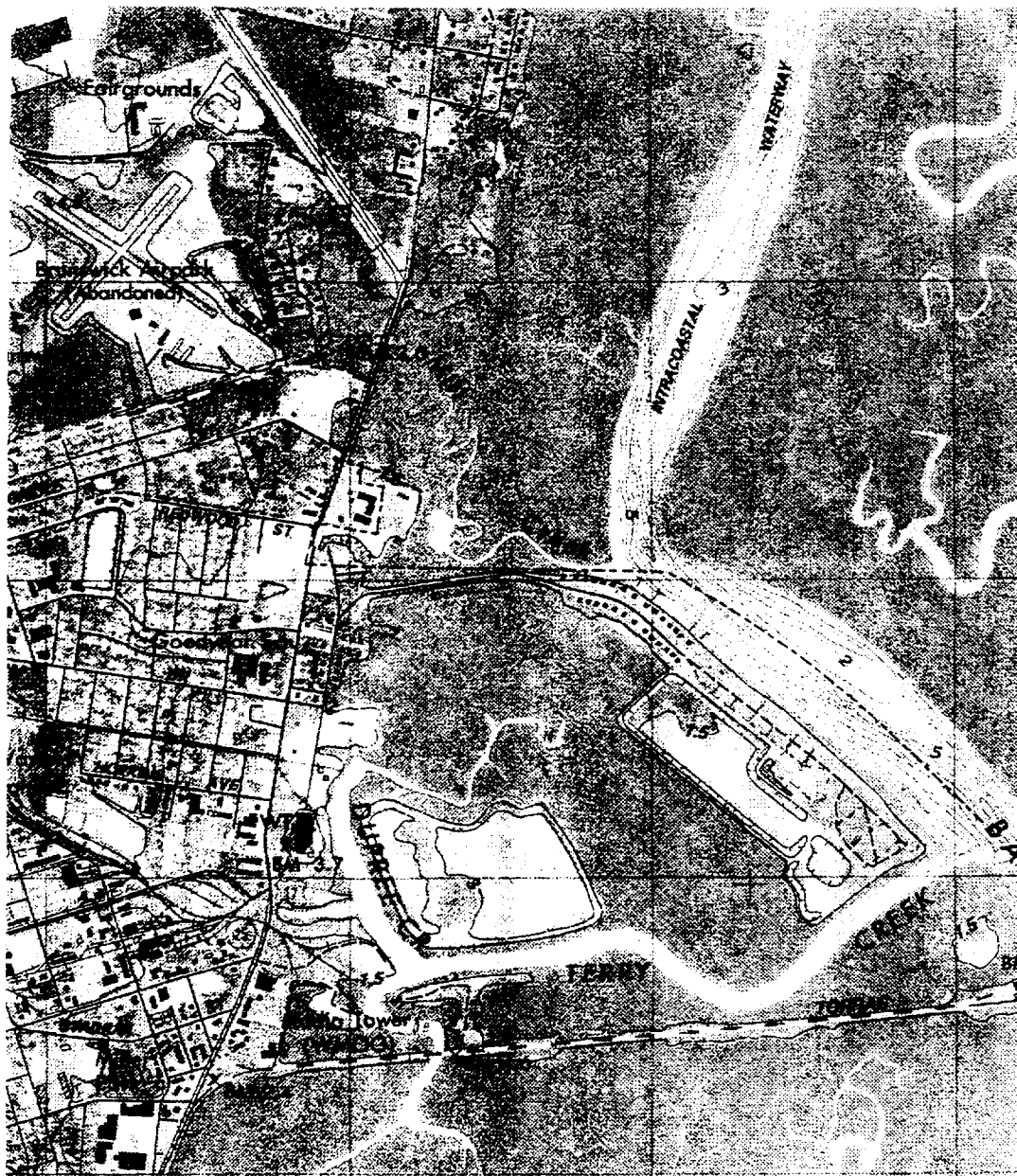
- 1) Sample Areas locations are shown in Figure 3.
- 2) All these samples will analyzed for toxaphene. Analyses of subsamples may occur according to Section 2.2.1.
- 3) Five of the sampling locations shown in the Dredge Spoil Area on Figure 3 will include analysis of water samples from porous-cup lysimeters. Permeability will be measured in separate samples collected in the berm samples using test method ASTM D-5084. These locations are not on Figure 3.
- 5) Quality assurance sample frequency is in accordance with USEPA Region 4 Standard Operating Procedures [USEPA, 1996], and includes: duplicate of 1 out of 20 samples, and one equipment rinsate blank per week of field activity.
- 6) Samples will be obtained from a depth of 1 to 2 ft.

FIGURES

LOCATION OF THE TERRY CREEK SITE BRUNSWICK, GEORGIA

2

0037



SOURCE: USGS BRUNSWICK EAST
7.5 MIN TOPO QUADRANGLE (1982)

1000 0 1000 2000 Feet



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ATLANTA, GEORGIA

FIGURE NO.	1
PROJECT NO.	GQ0270
DOCUMENT NO.	GA970848
FILE NO.	FIG1.APR




PROPOSED SAMPLING LOCATIONS TERRY CREEK SITE

LEGEND

- Transect
- Weir
- Terry Creek Proposed Sample Location
- Dredge Spoon Area Sample Point
- North Marsh Area Sample Point
- Dupree Creek Sample Point
- Terry Creek Sample Point
- Berm Sample Point
- Outfall Ditch Sample Point
- Sample Point with Livestake



 GEOSYNTEC CONSULTANTS ATLANTA, GEORGIA			
DATE	25 JULY 1997	SCALE	1"=600'
PROJECT NO.	300275	FIGURE NO.	1
DOCUMENT NO.	GA970848	FILE NO.	3870848.APR

AIRIAL PHOTOGRAPH DATED 1996

2
4
0003



AERIAL PHOTOGRAPH DATED 1994

PREVIOUS SAMPLING LOCATIONS

LEGEND

- Wet
- Previous Sampling Locations
- Surface Soil
- Soil Benthos
- Sediment
- Deep Sediment
- Bank Sediment

300 0 300 600 900 Feet



GeoSyntec Consultants

ATLANTA, GEORGIA

DATE	25 JULY 1997	SCALE	1"=600'
PROJECT NO.	GQ0270	FIGURE NO.	2
DOCUMENT NO.	GA970848	FILE NO.	SSWORK APR

Prepared for:

Hercules Incorporated

Hercules Plaza
1313 North Market Street
Wilmington, Delaware 19894-0001

**REVISION 0
QUALITY ASSURANCE
PROJECT PLAN**

**TERRY CREEK SITE
BRUNSWICK, GEORGIA**

Prepared by:



GEOSYNTEC CONSULTANTS

1100 Lake Hearn Drive, NE, Suite 200
Atlanta, Georgia 30342

Project Number GQ0270-02

August 1997

Revision 0

GeoSyntec Consultants

APPROVALS**Approval for GeoSyntec Consultants**

Project Manager

Signature: L. Minto Date: 15 August 1997

Project Quality Assurance/Quality Control Officer

Signature: Mary A. Redican Date: 15 August 1997

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1. INTRODUCTION

1.1 Terms of Reference

This Quality Assurance Project Plan (QAPP) was prepared by GeoSyntec Consultants (GeoSyntec) on behalf of Hercules Incorporated (Hercules) for the Terry Creek Site located in Brunswick, Georgia. This QAPP is a companion document to the Sampling and Analysis Plan (SAP) which is also prepared by GeoSyntec. These documents are being prepared at the request of the United States Environmental Protection Agency (USEPA), Region IV Emergency Response Group who is considering a removal action if unacceptable levels of toxaphene are found in at the Terry Creek Site.

This QAPP has been prepared based on the guidance and recommended format provided in the USEPA document "Model Quality Assurance Project Plan" [USEPA, 1991]. The USEPA Region IV document "Investigations and Standard Operating Procedures and Quality Assurance Manual [USEPA, 1996] was also used as guidance.

This QAPP was prepared by Mr. Charles A. Spiers, P.G. and it was reviewed by Dr. J.F. Beech, P.E. in accordance with GeoSyntec's peer review policy.

1.2 Site Description and History

Portions of Terry Creek and Dupree Creek initially were dredged by the Army Corps of Engineers (USACE) between 1938 and 1948. No dredging of these areas occurred again until the early 1970s. During the period in which dredging was not occurring, Hercules began to produce toxaphene, a pesticide, in its plant in Brunswick. Until production of this product was discontinued in 1980, some amounts of this material was contained in legal discharge from the plant. This discharge entered into Dupree Creek.

In 1972, with the approval of the USEPA and the US Fish and Wildlife Services and the State of Georgia, the USACE chose an area on the North side of Terry Creek at the confluence of Terry and Dupree Creeks in which to place dredge spoils from the

dredging of Terry and Dupree Creeks (i.e., the Dredge Spoil Area). Dredge spoils were placed in this area from 1972 until dredging was discontinued in the late 1980s. The Dredge Spoil Area and areas adjoining it are the subject of this report.

The primary disposal area used by the USACE at the Terry Creek Site is the Dredge Spoil Area. The dredge spoil in this area contains toxaphene. The Dredge Spoil Area is surrounded by a berm. Initially, a low-level berm was constructed from marsh sediment. Dredge spoil was placed within the berm and allowed to dewater by gravity drainage. As solids settled out of the dredge slurry, the resulting water was allowed to drain through three weirs back into Terry and Dupree Creeks. The dewatered dredge material was pushed up against the inside of the original low-level berm to form a higher inner berm. The dewatered dredge material was not pushed in the front of the original, low-level berm.

1.3 Project Background

In 1995, the USEPA Region IV conducted an Expanded Site Inspection (ESI) of the Terry Creek Site. A total of 45 samples were collected during the ESI that included: groundwater samples, surface and subsurface soil samples, surface water samples and sediment samples collected from Dupree and Terry Creeks and the Back River. Samples of soil and sediment collected in the dredge spoil area, as well as the sediments of Dupree Creek and Terry Creek, contained measurable concentrations of toxaphene (Figure 2).

Data about the Terry Creek Site have been gathered as a result of the ESI, previous sampling events conducted by Hercules, the USACE, the Brunswick Initiative and the State of Georgia Environmental Protection Division (GAEPD). The USEPA Region IV and Hercules are considering a removal action of contaminated soil and sediment in and around the dredge spoil area. USEPA is currently reviewing available data. As part of its evaluation, USEPA has requested additional samples of soil and sediment from the Terry Creek Site be collected and analyzed.

Hercules has committed to further sampling. A sampling and analysis plan (SAP) has been prepared for the Terry Creek site [GeoSyntec, 1997] which addresses sample

collection, establishing sample locations, equipment decontamination, and general procedures and guidelines for field documentation.

1.4 DQO Analytical Levels

Data Quality Objective (DQO) analytical levels are established to ensure that the data collected are sufficient and of adequate quality for their intended uses. Five levels of data quality are recognized by the USEPA:

- Level I data are typically collected using portable field equipment and are used for gross engineering determinations and for health and safety.
- Level II data are not subject to strict QA/QC procedures. These data may be used for quantitative screening or to determine sampling locations.
- Level III data are generated by laboratories using USEPA procedures and may be used for engineering evaluation in feasibility studies or remedial design.
- Level IV data are generally used for risk assessment, remedial investigations and feasibility studies.
- Level V data include unconventional methods or parameters.

All soil, sediment, and dredge material samples will be chemically analyzed by En Chem, Inc., (En Chem) of Madison, Wisconsin. Analytical laboratory data validation will be provided by Environmental Standards, Inc. of Valley Forge, Pennsylvania. The Level IV DQO will be used for this project. Geotechnical testing will be performed by GeoSyntec.

2. PROJECT ORGANIZATION AND RESPONSIBILITIES

2.1 Overview

This section describes the project organization and the responsibilities of the key project personnel for maintaining proper QA/QC for the sampling and analysis of environmental media as part of the investigation. The organizational structure is presented in Figure 2.

2.2 Hercules Incorporated

The Hercules project coordinator's responsibilities include oversight and technical input of the removal action sampling activities. The activities also include oversight of the analytical laboratory and validation of the analytical data. Mr. Timothy D. Hassett is the Project Coordinator for Hercules. Mr. Wayne Quinn will be the Site Field Engineer for Hercules.

2.3 GeoSyntec Consultants

2.3.1 Principal-in-Charge

The Principal-in-Charge will direct the project management and peer review team. He will be responsible for ensuring that: (i) the project is appropriately staffed and all necessary resources are available to complete the project; and (ii) the project strategy, goals, and schedule are consistently achieved in all tasks. The Principal-in-Charge also provides a supplemental line of communication between Hercules and GeoSyntec.

2.3.2 Corporate Health and Safety Officer

The GeoSyntec Corporate Health and Safety Officer is responsible for developing and overseeing the corporate health and safety program. The GeoSyntec Corporate Health and Safety Officer will provide direction to the GeoSyntec Project Manager and/or the Project Health and Safety Officer, as necessary, on issues of health and safety.

2.3.3 GeoSyntec Project Manager

The GeoSyntec Project Manager will be the primary point of contact between the Hercules project coordinator and the project team and will be responsible for project planning, scheduling, budgeting, staffing, cost control, fulfilling the proposed scope of services, and work products. Through these activities he will ensure key technical issues are properly evaluated and the client's best interests are addressed at each stage of the project.

2.3.4 GeoSyntec Quality Assurance/Quality Control (QA/QC) Officer

The GeoSyntec Quality Assurance/Quality Control (QA/QC) Officer's responsibilities include management of all aspects of quality control and quality assurance as required by the Quality Assurance Project Plan. The GeoSyntec QA/QC Officer is also responsible for reviewing, on behalf of GeoSyntec the data validation report from Environmental Standards.

2.3.5 GeoSyntec Project Health and Safety Officer

The GeoSyntec Project Health and Safety Officer will provide overall health and safety services for the project team. The GeoSyntec Project Health and Safety Officer will report to the GeoSyntec Project Manager and will ensure that all health and safety requirements of the Occupational Health and Safety Administration (OSHA) and CERCLA are followed.

2.3.6 GeoSyntec Site Health and Safety Officer

The GeoSyntec Site Health and Safety Officer's (SHSO) responsibilities include evaluating the appropriate level of personal protective equipment. The SHSO will also be responsible for field team operations and safety.

2.3.7 GeoSyntec Field Manager

The GeoSyntec Field Manager's responsibilities include implementing the sampling activities. If the Field Manager serves as Site Health and Safety Officer, he may also assume the responsibility of evaluating the appropriate level of personal protective equipment and be responsible for field team operations and safety.

The Field Manager will work closely with the GeoSyntec QA/QC Officer to ensure that QA data is collected and coordinated properly. All field technicians will report to the Field Manager in accordance with the Manufacturer's recommendations. The Field Manager is responsible for ensuring all equipment is calibrated and test results are properly recorded on field logs.

2.3.8 Analytical Laboratory

The Analytical Laboratory's responsibilities include chemical analysis of samples and implementation of QA/QC procedures as required by this QAPP and as specified in the analytical methods. Analytical services will be provided by En Chem. En Chem's Quality Assurance Manual (QAM) is included in Appendix A. En Chem's Standard operating Procedure (SOP) for Toxaphene Soil Extraction and Analysis is included in Appendix B. The analytical method for the determination of toxaphene is included in Appendix C. Where this method differs from the En Chem's SOP, the specific instructions in Appendix C must be followed. The specific guidance agreed upon by USEPA Region IV is included in Appendix C. Geotechnical testing will be provided by GeoSyntec.

2.3.9 Geotechnical Laboratory

Geotechnical testing will be provided by GeoSyntec's GeoEnvironmental Laboratory (GEL) in Alpharetta, Georgia. The Standard Operating Procedures for the GEL are included in Appendix D.

3. QA OBJECTIVES FOR MEASUREMENT OF DATA

3.1 Overview

The overall QA objectives are to develop and implement procedures for field sampling, chain-of-custody, laboratory analysis, and reporting that will provide results which are legally defensible. The purpose of this section is to address the specific quantitative QA objectives of accuracy, precision, and completeness, along with the qualitative QA objectives of representativeness, and comparability.

3.2 Quality Assurance Objectives for Chemical Analysis of Environmental Media

The definitions for precision, accuracy, completeness, representativeness, and comparability are as follows:

- precision - a measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions, usually expressed in terms of relative percent difference;
- accuracy - the degree of agreement of a measurement with an accepted reference or true value;
- completeness - the amount of valid data obtained from a measurement system compared to the amount that was expected under normal conditions; and
- representativeness - the selection of analytical methods and sampling protocols and locations such that results are representative of the media being sampled (i.e., water soil, air, etc.) and conditions measured;
- comparability - expresses the confidence with which one data set can be compared with another.

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Analytical precision will be evaluated for analytical methods by comparing the QC criteria stipulated in the method standard operating procedures (SOP) to the results from laboratory matrix spike/matrix spike duplicate samples and field duplicate samples.

The accuracy of the analytical data will be assessed by examining the results obtained from the required laboratory QA/QC samples. Data will be qualified in accordance with the appropriate USEPA functional guidelines. The methods for calculating the QA/QC sample precision and accuracy are provided in Section 12 of this document.

Representativeness will be evaluated based on the analysis and interpretation of the results of an appropriately defined number of QC samples. The number and type of internal and external QC samples are defined in Section 8 of this document.

The completeness goal for this program is greater than 80 percent. This completeness goal is considered adequate to meet the data quality objectives for this site based on sampling design plans.

4. SAMPLING PROCEDURES

4.1 Overview

This section discusses the standard practices and procedures utilized during the field operations to ensure the collection of representative samples. The collection of representative samples depends upon:

- ensuring that the sample taken is representative of the material or medium being sampled
- using proper sampling, sample handling, preservation, and quality control techniques;
- properly identifying the collected samples and documenting their collection in permanent field records (field log books, chain-of-custody records); and
- maintaining sample chain-of-custody.

4.2 Sample Containers, Preservatives, and Holding Times

Sample containers, preservatives and holding times for each of the parameters to be measured are identified in Table 1. All sample containers for samples submitted for laboratory analyses will be provided by the contract laboratory. Containers will be new, pre-cleaned, or pre-baked as appropriate.

Sample preservation other than temperature control is not required for soil or sediment samples. Sample containers will be sealed with custody tape in the field by sampling personnel.

Solid samples (e.g., soil and sediment) collected for the analysis of toxaphene can be stored for up to 14 days prior to extraction. The sample extracts must be analyzed within 40 days of extraction. Aqueous samples must be extracted within 7 days of collection and extracts analyzed within 40 days of extraction.

4.3 Sample Packing Shipping and Documentation

4.3.1 Sample Packing

The sediment and soil samples obtained in the removal action sampling will be placed in shipping coolers with enough ice to maintain a temperature of 4°C and with sufficient bubble pack to prevent breakage during shipping. All samples in a shipping container will be listed on the chain-of-custody form enclosed in the shipping container. Once the samples are securely packaged, the container will be sealed with tape and several custody seals will be placed over the top edge so that the container cannot be opened without breaking the custody seal.

4.3.2 Sample Shipping

All samples will be shipped via courier service (Federal Express, Airborne, etc.) and will include a separate, signed chain-of-custody form enclosed in the shipping container. A shipping document for the courier service will be completed for each shipment. Shipments will occur approximately on a daily basis to minimize the storage of samples at the site and to maximize the holding time period available to the laboratory for sample extraction and analysis.

4.3.3 Sample Documentation

4.3.3.1 Overview

Documents recording sampling events will include a daily field activity log, health and safety log, field sampling logs, sample labels, and chain-of-custody seals and forms. The information to be recorded for each sampling event is discussed in the "*Sampling and Analysis Plan, Terry Creek Site, Brunswick, Georgia*", (SAP) [GeoSyntec, 1997].

4.3.3.2 Sample Labels

Each sample bottle will be labeled with the following information: date and time of sample collection, sample number, analyte(s), preservative, project and task number, and sampler's initials. Indelible ink will be used to record information on the sample label and the sample identification scheme discussed in the SAP will be followed [GeoSyntec, 1997].

4.3.3.3 Custody Seals

Custody seals will be used when a sample shipment is picked up by the laboratory or sent to the laboratory by overnight courier. Signed and dated custody seals will be attached to each sample jar in such a way that it is necessary to break the seal to open the jar. Signed and dated custody seals will be attached to the top of the shipping container in such a way that it is necessary to break the seal to open the container. Custody seals ensure that any tampering during transportation will be detected by the receiving laboratory.

4.3.3.4 Chain-of-Custody Forms

Chain-of-custody forms provide the documentation to trace sample possession from the time of sample collection until receipt by the laboratory. One chain-of-custody form will be filled out for each cooler or shipping container and will list all the samples contained in the cooler or container. An example chain-of-custody form is presented in Appendix A of this QAPP. One copy of the completed form will be placed in a plastic bag taped to the inside lid of the shipping container and one copy will be kept with the project files.

5. SAMPLE CUSTODY PROCEDURES

5.1 Overview

The possession of samples will be traceable from the time that they are collected until they are disposed by the analytical laboratory. The following custody procedures provide this means of sample tracking.

5.2 Sample Custody

A sample or other physical evidence is in custody if:

- it is in the field investigator's, transferee's, or lab technician's actual possession; or
- it is within the field investigator's, transferee's, or lab technician's view, after being in his/her physical possession; or
- it was in the field investigator's, transferee's, or lab technician's physical possession and then he/she secured it to prevent tampering; or
- it is placed in a designated secure area.

5.3 Chain-of-Custody Record

The field chain-of-custody record is used to record the custody of all samples or other physical evidence collected and maintained. This form shall not be used to document the collection of split samples where there is a legal requirement to provide a receipt for samples. The chain-of-custody record also serves as a sample logging mechanism for the analytical laboratory's sample custodian.

The type of information to be supplied on the field chain-of-custody record includes:

- the project number;
- the project name;
- all samplers and/or the sampling team leader's signature;
- the sampling station number, date, and time of sample collection, grab or composite sample designation, and the sampling location;
- container type and the total number of sample containers;
- sample volumes,
- preservative(s), if any, and
- analyses to be performed.

The chain-of-custody record is a serialized document. Once the record is completed, it becomes an accountable document and must be maintained in the project file. An example chain-of-custody record is presented in the En Chem QAM (Appendix A).

5.4 Field Custody

The Field Manager is responsible for the proper handling and custody of the samples collected until they are properly and formally transferred to another person or facility. The Field Manager will ensure that all samples (i) have a sample label completed for each sample, using waterproof, non-erasable ink; and (ii) are documented in bound field log books or on field sampling forms.

5.5 Transfer of Custody and Shipment

All samples will be accompanied by the chain-of-custody record. The original record will be placed in a plastic bag inside the secured shipping container if samples are shipped. A copy of the chain-of-custody record will be retained by the field

sampling leader. When transferring the possession of samples, the individual receiving the samples will sign, date, and note the time that he/she received the samples on the chain-of-custody record. This chain-of-custody record documents transfer of custody of samples from the field investigator to another person or to the laboratory. The original record will be sent to the GeoSyntec QA/QC officer after the laboratory analyzes the sample. This copy will become a part of the project file.

Samples will be properly packaged for shipment and delivered or shipped to the designated laboratory for analyses. Shipping containers shall be secured by using strapping tape and custody seals. The custody seals shall be placed on the container so that it cannot be opened without breaking the seals. The seal shall be signed and dated by the field investigator.

5.6 Document Control

The term document control, as it applies to field investigations, refers to the maintenance of project files. The following documents shall be placed in the project file:

- a copy of the approved work plan and supported plans;
- original chain-of-custody records, bound field log books, and field forms;
- pertinent records obtained during the investigation;
- a complete copy of the analytical data and memorandum transmitting analytical data;
- official correspondence received by or issued by the USEPA relating to the investigation including records of telephone calls;
- one copy of the final report and transmittal memorandum(s); and
- any other relevant documents related to the investigation or follow-up activities.

6. CALIBRATION PROCEDURES AND FREQUENCY

6.1 Field Instruments

Field testing instruments are anticipated for use during sediment sampling of the streams near the Dredge Spoil Area. Preferably, a single instrument which has the capability to measure pH, conductivity, salinity, dissolved oxygen, and temperature will be used. However, multiple instruments are acceptable. The instrument or instruments will be calibrated in accordance with the manufacturer's recommended procedures and calibration standard solutions. Calibration frequency will be a minimum of daily unless more frequent calibration is recommended by the instrument manufacturer.

6.2 Laboratory Instruments

Calibration of laboratory equipment will be based on approved written procedures. Specific calibration procedures for laboratory equipment are described in En Chem's QAM, which is presented in Appendix A. Records of calibration, repairs, or replacement will be filed and maintained by the designated laboratory personnel performing quality control activities. These records will be filed at the location where the work is performed and will be subject to QA audit.

7. ANALYTICAL PROCEDURES

7.1 Introduction

Analytical procedures applied to samples obtained from the site may include field methodologies, SW-846, and other USEPA-approved laboratory analytical procedures.

7.2 Laboratory Analyses

A summary of the specific methods that may be used during the sampling program is provided in Table 2. Accompanying each method are reporting limits, where applicable, that are achievable when little or no interferences are present.

En Chem has developed standard operating procedures (SOPs) for sample extraction prior to analysis for PCB's and organochlorine pesticides, such as toxaphene. The SOP utilized varies depending on the range of toxaphene concentrations in the samples. Descriptions of the En Chem sample extraction SOPs are provided in Appendix B. However, En Chem must follow the specific methods provided in Appendix C of this QAPP.

All samples will be analyzed for toxaphene using USEPA SW-846 Method 8081. The method of analysis and the extraction methods, which were agreed upon by the USEPA Region IV laboratory, are provided in Appendix C of this QAPP.

7.3 Field Analyses

Field parameters of pH, conductivity, salinity, dissolved oxygen and temperature will be measured in water during sediment sampling of the streams near the Dredge Spoil Area. The parameters will be measured using calibrated meters, and the results of these measurements will be recorded in the field log notebooks. Calibration solutions will be used in the field in accordance with the instrument manufacturer's instructions. Calibration will take place each day for each meter and will be recorded in the field notebooks.

8. INTERNAL QUALITY CONTROL CHECKS

8.1 Overview

Internal QC procedures and samples are designed to ensure and document the overall quality of data. QC procedures and QC samples are described below.

8.2 QC Procedures

QC procedures include field instrumentation and those conducted by En Chem. QC procedures for field instrumentation measurements will include: (i) using fresh standard calibration solutions; (ii) using multiple standard solutions which bracket the range of anticipated measurements (i.e., pH range of 5 to 7); and verifying that the calibration solutions are appropriate for the environmental conditions (i.e., high salinity).

8.3 QC Samples

Field sampling procedures require the preparation and submittal of two types of QC samples from the field:

- *Equipment Rinsate Blanks* - Equipment rinsate blanks are prepared in the field to verify that a sampling device (e.g., trowel or auger) is free from contamination. A sampling device is rinsed with distilled or deionized water, and the rinsings are transferred to the appropriate sample bottles, preserved, and submitted to the laboratory for analysis. Rinsate blanks are prepared at the outset of field sampling and once a week thereafter.
- *Blind Duplicates* - Two sets of samples from a source are prepared, labeled with unique sample numbers, and submitted to the laboratory without identifying the samples as duplicates. One blind duplicate will be prepared for every 20 environmental samples collected for each matrix type.

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Laboratory QC procedures are outlined in the En Chem's QAM in Appendix A. Note, matrix spike/matrix spike duplicate samples and all laboratory control spikes (LCS) samples will be spiked with toxaphene.

9. DATA VALIDATION

9.1 Overview

This section provides a summary of the methods to be used to verify that the collected data meet the identified data quality objectives. The laboratory data, geotechnical data, and field testing data will then be checked for completeness and will undergo final validation and data review by the QA/QC Officer. The data review procedures for the QA/QC Officer are discussed in Section 9.3.

9.2 Laboratory Data Quality Review and Validation

The analytical laboratory will perform in-house analytical data reduction and QA review in accordance with a multiple level process as follows:

- analytical review performed by the bench analyst;
- technical review performed by a lab manager or team leader;
- QA review on selected data performed by a quality assurance specialist; and
- diskette deliverable review by a systems specialist (optional).

Specific data review procedures are described in the laboratory QAM which is presented in Appendix A. The laboratory will notify Hercules of any data that may be qualified as preliminary, unacceptable, or having other limitations with respect to data quality.

9.3 Review and Validation of Data Received From the Laboratories

GeoSyntec will review field data and geotechnical laboratory data. Environmental Standards, Inc. will review analytical reports received from the laboratory for appropriateness of the field and laboratory testing methodologies, precision, accuracy, comparability, and completeness.

Data review will generally include the following steps:

- verifying completeness of data;
- verifying sample custody and verifying that samples were appropriately collected and preserved in the field and laboratory;
- verifying that samples were analyzed within appropriate sample holding times;
- verifying appropriateness of field and laboratory test methods;
- comparing data to objectives for precision, accuracy, and comparability;
- evaluating field QA/QC sample data, such as blanks and duplicates;
- evaluating laboratory QA/QC sample data, such as duplicates, spikes, and blanks;
- checking for discrepancies (such as transcription and calculation errors) and data outliers;
- discussing identified discrepancies with the field personnel or laboratory, as appropriate, to clarify the reason for the discrepancy and to formulate a course of action; and
- accepting, rejecting, or qualifying data with respect to the acceptance criteria.

When data discrepancies and outliers are identified that could potentially result in rejection or qualification of data, corrective action steps will be taken. Specific data validation procedure criteria to be followed are described in the “*National Functional Guidelines for Organic Data Review*” [USEPA, 1994a].

The following data analysis methods may be used during data validation to assist in the data quality review, and after data validation to assist in data interpretation:

- preparation of data summary tables;
- mapping the aerial extent of specific chemicals;

- preparing plots of chemical concentrations over time at specific locations; and
- conducting statistical analysis of data, including: (i) calculation of means, modes, standard deviations, and/or coefficients of variation; and (ii) trend analysis.

All data points rejected during the data validation stage will be deleted from the final tables, plots, and other data analysis products. Qualified data, will be included and data usability assessment will be performed on all data. All tables, plots, and other data analysis products will be checked and reviewed. Data review summaries, including data quality summaries, will be included in the final report.

9.4 Data Deliverables

Data deliverables from the analytical laboratory will consist of the following items:

- Case Narrative;
- Laboratory Final Reports;
- Surrogate Recovery Summary;
- Matrix Spike/Matrix Spike Duplicate Recovery Summary;
- Method Blank Summary;
- Laboratory Control Sample (LCS) Recovery Summary;
- Initial Calibration Summary (GC Method Printout);
- Continuing Calibration Summary;
- Analytical Sequence Printout;
- Chromatographs and Quantification Reports for all Samples, Standards, and QC Samples;

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- Copies of Extraction Log Pages; and
- Copies of Chain-of-Custody Document.

For consistency and ease of review, the data deliverables will be organized in the same manner. The arrangement will be as follows:

- Sample Narrative;
- Final Reports;
- QC Summary Information;
- Analytical Sequence Printout(s);
- Sample Raw Data (arranged by sample number);
- Instrument Calibration Data (in chronological order);
- Raw QC Data;
 - Blanks,
 - LCS,
 - MS/MSD, and
- Extraction Logbook Pages; and
- Chain-of-Custody Documents.

10. PERFORMANCE AND SYSTEM AUDITS

10.1 Overview

Performance and system audits for sampling and analyses operations consist of on-site review of field and laboratory QA systems and on-site review of equipment for sampling, calibration, and measurement. The audits are designed to evaluate the capability and performance of project and subcontractor personnel, activities, and documentation.

10.2 Field Performance/System Audit(s)

The GeoSyntec QA/QC Officer or an appointed non-project participating representative will make one or more non-scheduled visits to the selected project site to observe the performance of the field operations team during all phases of field activities (monitoring well installation, ground-water sampling, etc.). During this same period of time, a systems audit of field operations personnel may also be performed. The audits will be performed and assessed in accordance with guidelines set forth in the "*A Compendium of Superfund Field Operations Methods*" [USEPA, 1987].

10.3 Laboratory System Audit

A laboratory systems audit may be conducted prior to and/or during the course of the project. These audits are designed to ensure that the systems and operational capabilities of the laboratory are maintained and methodology and quality control measures for the project are being followed as specified by the QAPP and the laboratory QAM. All audits will be performed and assessed in accordance with guidelines set forth in the "*A Compendium of Superfund Field Operations Methods*" [USEPA, 1987].

11. MAINTENANCE PROCEDURES

11.1 Field Equipment/Instruments

Specific preventive maintenance procedures to be followed for field equipment are those recommended by the manufacturer. Backup instruments and equipment will be available on-site or within one-day shipment to avoid delays in the field schedule.

11.2 Laboratory Instruments

The analytical laboratory, En Chem, has SOPs for preventive maintenance for each measurement system and required support activity. En Chem will document all maintenance activities in log books to provide a history of maintenance records. A more detailed description of the laboratory maintenance procedures is provided in the laboratory QAM, presented in Appendix A.

12. SPECIFIC ROUTINE PROCEDURES TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

12.1 Field Measurements

Accuracy and precision were defined in Section 3. Accuracy of the field measurements will be assessed using daily instrument calibration, calibration check, and analysis of blanks. Precision will be assessed on the basis of reproducibility by multiple reading of a single sample. Data completeness will be calculated using Equation 12-1.

$$\% \text{ Completeness} = \frac{\text{Valid Data Obtained}}{\text{Total Data Planned}} \times 100 \quad (\text{Equation 12-1})$$

12.2 Laboratory Data

Laboratory results will be assessed for compliance with required precision, accuracy, completeness, and sensitivity as described in the following sections.

12.2.1 Precision

Precision of laboratory analysis will be assessed by comparing the analytical results between matrix spike/matrix spike duplicate (MS/MSD) for organic analysis, and field and laboratory duplicate analyses. The relative percent difference (% RPD) will be calculated for each pair of duplicate analyses using the Equation 12-2

$$\%RPD = \frac{S - D}{(S + D) / 2} \times 100 \quad (\text{Equation 12-2})$$

where: S = first sample value (original or MS values); and
D = second sample value (duplicate or MSD value).

12.2.2 Accuracy

Accuracy of laboratory results will be assessed using the analytical results of matrix spike/matrix spike duplicate, laboratory control, and surrogate recovery samples. The percent recovery (%R) of matrix spike samples will be calculated using Equation 12-3:

$$\%R = \frac{A - B}{C} \times 100 \quad (\text{Equation 12-3})$$

where: A = the analyte concentration determined experimentally from the spiked sample;
B = the background level determined by a separate analysis of the unspiked sample; and
C = the amount of the spike added.

12.2.3 Completeness

Completeness was defined in Section 3. Data completeness will be evaluated during data validation, and the resulting information will be used to determine the completeness of analyses. Overall criteria for data completeness will be determined and compared to project DQOs presented in Section 3.0 of this QAPP.

12.2.4 Sensitivity

The achievement of method detection limits depends on instrument sensitivity and matrix effects. Therefore, it is important to monitor the instrument sensitivity to ensure data quality through constant instrument performance. The instrument sensitivity will be monitored through the analysis of method blank, calibration check, and laboratory control samples, etc., as specified in the laboratory QAM, presented in Appendix A.

13. CORRECTIVE ACTION

13.1 Overview

The purpose of this section is to outline the procedures for implementing and documenting corrective actions, and to define the responsibilities of all appropriate personnel.

13.2 Field Corrective Actions

Technical staff and project personnel will be responsible for reporting all suspected technical or QA nonconformances or suspected deficiencies of any activity or issued document by reporting the situation to the Field Manager. The Field Manager will be responsible for assessing the suspected problems in consultation with the QA/QC Officer on making a decision based on the potential for the situation to impact the quality of the data. If it is determined that the situation warrants a reportable nonconformance requiring corrective action, then a nonconformance report will be initiated by the Field Manager.

The Field Manager will be responsible for ensuring that corrective action for nonconformances are initiated by:

- evaluating all reported nonconformances;
- controlling additional work on nonconforming items;
- determining disposition or action to be taken;
- maintaining a log of nonconformances;
- reviewing nonconformance reports and corrective actions taken; and
- ensuring nonconformance reports are included in the final site documentation in project files.

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If appropriate, the Field Manager will ensure that no additional work that is dependent on the nonconforming activity is performed until the corrective actions are completed.

Corrective actions will be implemented and documented in the field log book. No staff member will initiate corrective action without prior communication of findings through the proper channels.

13.3 Laboratory Corrective Action

Corrective actions are required whenever an out-of-control event is noted. The action taken is dependent on the analysis and the event. Laboratory corrective actions will be implemented according to the procedures described in En Chem's QAM.

14. QUALITY ASSURANCE REPORTS TO MANAGEMENT

Analytical results for samples analyzed during the field investigation will be summarized in a laboratory validation review report and submitted to the Hercules and GeoSyntec Project Managers following QA/QC review.

Upon completion of the project, a report will be submitted that will contain a discussion of QA/QC evaluations summarizing the quality of the data collected and used for each activity of the project. The objective of the QA/QC evaluations will be to ensure that the data are representative of site conditions and sufficient in quality and quantity to support the field activities.

15. REFERENCES

GeoSyntec Consultants, "Sampling and Analysis Plan, Terry Creek Site, Brunswick, Georgia", 1997a.

U.S. Environmental Protection Agency, "Test Methods for Evaluating Solid Wastes", SW-846, 3rd Edition, September 1986.

U.S. Environmental Protection Agency, Region V, "Model Quality Assurance Project Plan", May 1991.

U.S. Environmental Protection Agency, "A Compendium of Superfund Field Operations Methods", December 1987.

U.S. Environmental Protection Agency, "National Functional Guidelines for Organic Data Review", February, 1994a.

U.S. Environmental Protection Agency, "National Functional Guidelines for Inorganic Data Review", February, 1994b.

U.S. Environmental Protection Agency, Region IV, "Investigations and Standard Operating Procedures and Quality Assurance Manual", May 1996.

TABLES

TABLE 1
SAMPLE CONTAINERS, PRESERVATIVES AND HOLDING TIMES
TERRY CREEK SITE
BRUNSWICK, GEORGIA

Parameter	Matrix	Preservative	Holding Time	Container Size
Toxaphene	soil/sediment, sludge, dredged materials	Cool, 4°C	Extract within 14 days, and analyze extract within 40 days	500-mL widemouth amber glass container with Teflon- lined lid
Toxaphene	water	Cool, 4°C	Extract within 7 days and analyze extract within 40 days	1-L widemouth amber glass container with Teflon-lined lid
Total Organic Carbon	soil/sediment	Cool, 4°C	Analyze within 28 days	500-mL Plastic Container
Total Suspended Solids	water	Cool, 4°C	7 days	500-mL Plastic Container

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TABLE 2

**ANALYTICAL METHODS AND REPORTING LIMITS
TERRY CREEK SITE
BRUNSWICK, GEORGIA**

Parameters	Method⁽¹⁾	Soil Reporting Limits	Water Reporting Limits
Toxaphene	SW-846, 8081 ⁽¹⁾	0.17 mg/Kg	5 mg/L
Total Organic Carbon	SW-846, 9060 ⁽²⁾	100 mg/Kg	
Total Suspended Solids	Standard Methods 2540 ⁽³⁾		5 mg/L

Notes:

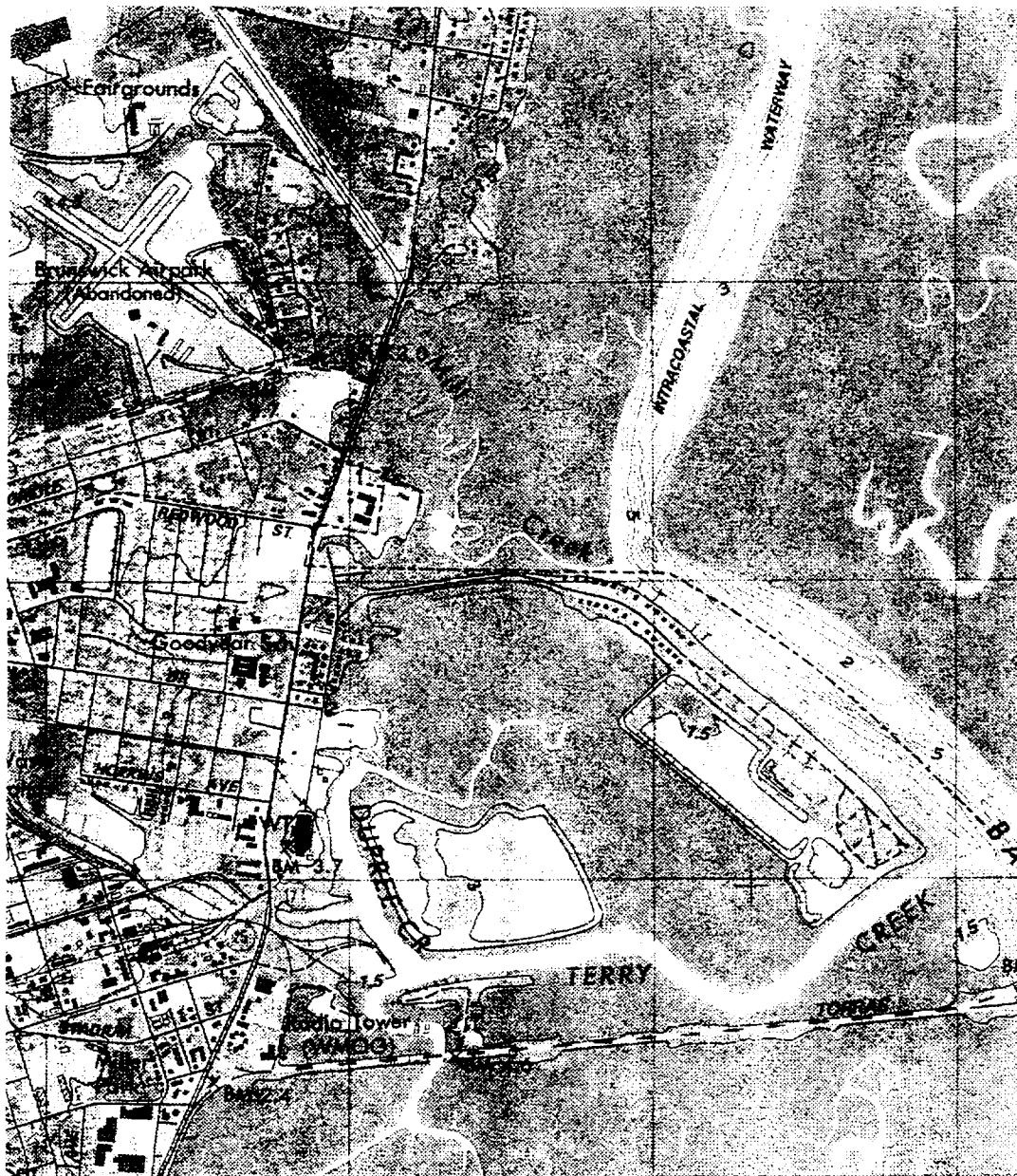
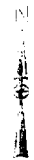
1. The analytical method to be used will follow the specific guidance agreed upon by USEPA Region IV.
2. EPA 9060 modified for soil.
3. Standard Methods for the Examination of Water and Waste Water, 18th Edition.

FIGURES

LOCATION OF THE TERRY CREEK SITE BRUNSWICK, GEORGIA

24

0005



SOURCE: USGS BRUNSWICK EAST
7.5 MIN TOPO QUADRANGLE (1982)

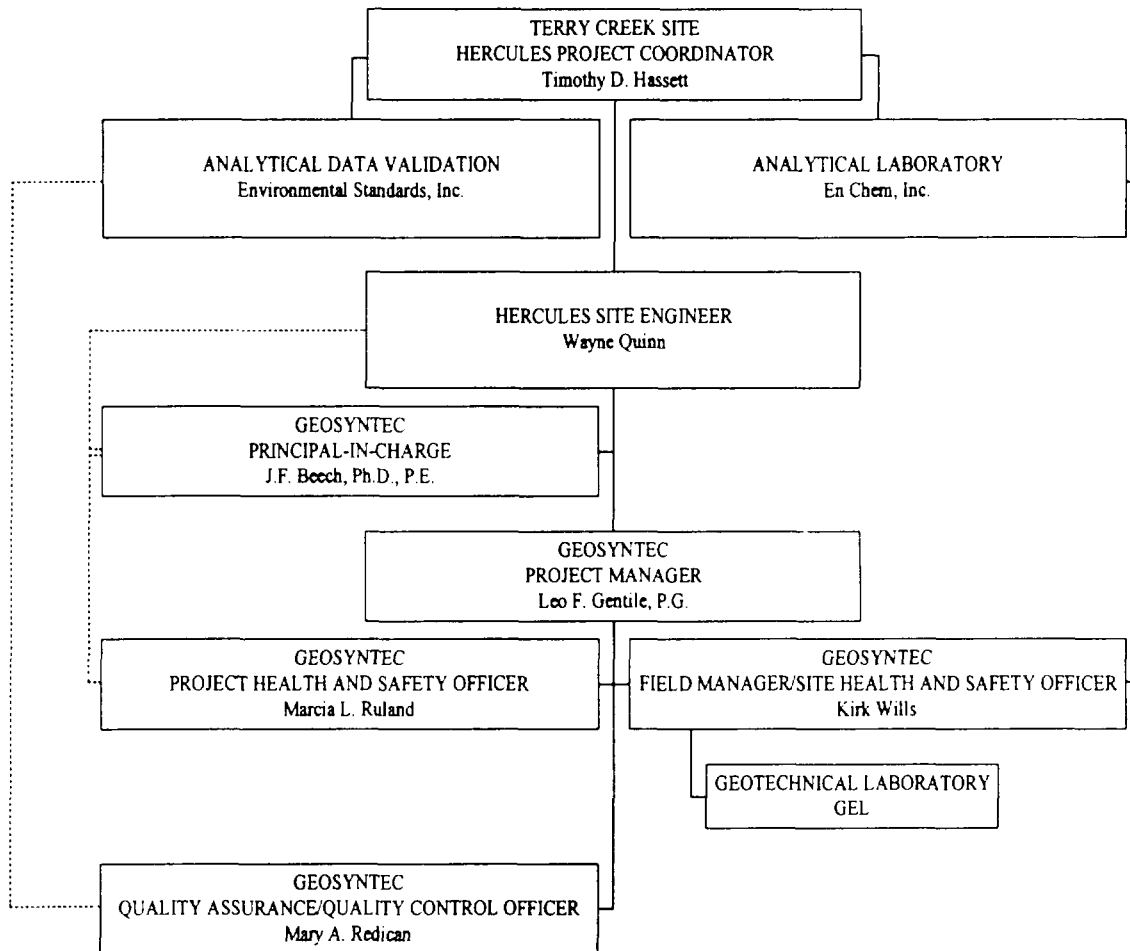
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GEOSYNTEC CONSULTANTS
ATLANTA, GEORGIA

FIGURE NO.	1
PROJECT NO.	GQ0270
DOCUMENT NO.	GA970859
FILE NO.	FIG1.APR

FIGURE 2
PROJECT ORGANIZATIONAL STRUCTURE
TERRY CREEK SITE
BRUNSWICK, GEORGIA



APPENDIX A
QUALITY ASSURANCE PLAN
EN CHEM, INC.



... chemistry for the environment

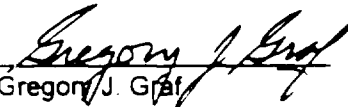
**EN CHEM - MADISON
QUALITY ASSURANCE MANUAL**

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STATEMENT OF MANAGEMENT POSITION

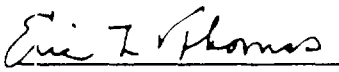
En Chem, Inc., is committed to producing analytical work of the highest quality to meet the needs of their clients and to assist in complying with all regulatory requirements.

This manual shall serve as a statement of the Company's quality assurance policies. Adherence to the procedures listed in this manual shall be the responsibility of all En Chem Laboratory employees. Laboratory management shall be responsible for seeing that the principles and practices outlined in the manual are followed.



Gregory J. Graf
Quality Assurance Officer

9-5-96
Date



Eric L. Thomas
Laboratory Manager

9/5/96
Date

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1.0 INTRODUCTION

The purpose of the En Chem Laboratory Quality Assurance Program is to verify that analytical data provided by the laboratory are of good quality and meet all pertinent regulatory requirements. This requires a comprehensive program which controls:

- Sample receipt
- Sample handling
- Sample log-in
- Sample preservation
- Sample processing
- Sample analysis
- Equipment maintenance
- Equipment calibration
- Data calculation
- Data reporting
- Records maintenance
- Data review
- Management responsibilities

This manual is intended to be a summary of the quality assurance procedures used in this laboratory.

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1.1 QUALITY ASSURANCE OBJECTIVES

The goal of any laboratory quality assurance program is the production of laboratory data of known quality.

This requires a comprehensive and effective quality control program to measure and verify laboratory performance, and the use of approved or proven methods to produce data that are accurate, precise, and complete. In addition, the system must identify factors which adversely affect quality and provide for corrective action where required. The system must also provide for the maintenance of records relating to sample submittal and the production of laboratory data.

Specifically, the quality assurance program must address the following topics:

- Specifications for supplies and instrumentation
- Sample receipt and storage
- Laboratory chain-of-custody
- Sources of laboratory methods
- Instrument calibration and preventive maintenance
- Statistical analysis of quality control data
- Data validation and reporting
- Laboratory records
- Corrective action
- Staff training
- Laboratory audits

1.2 QUALITY ASSURANCE DOCUMENTS

- There are several types of quality assurance documents.
- The Quality Assurance Manual provides the overall policy for the laboratory.

- Standard Operating Procedures (SOP's) are detailed instructions outlining a specific routine task performed in the laboratory.
- Project Specific Manuals may be prepared where a project requires unique or different quality assurance requirements or when they are required by regulatory agencies. These documents are frequently called Quality Assurance Project Plans (QAPP's).

1.3 DOCUMENT CONTROL

All of the Quality Assurance Documents listed above shall be approved and controlled documents as spelled out in this paragraph. The Quality Assurance Manual shall require the approvals of the Quality Assurance Officer and the Laboratory Manager before changes are issued. SOP's shall be reviewed by the Quality Assurance Officer and approved by the Laboratory Manager. QAPP's require the approval of individuals at all levels of the project. Documents shall be signed and dated by the necessary individuals before issuance.

The Quality Assurance Manual and the SOP's shall be numbered, and distribution lists shall be maintained so that all appropriate individuals receive updates. Revisions shall require the same signature levels as the originals and shall be consecutively numbered. All revisions shall be accompanied by a receipt which shall be signed and returned to signify that the revision has been received and placed in the proper location. Unnumbered copies of quality assurance documents may be issued to parties outside of En Chem, Inc. Where required, a numbered copy may be issued to parties outside of the Company. This numbered copy shall be updated the same as internal copies but must be returned to En Chem, Inc. when the need for the document no longer exists.

2.0 LABORATORY ORGANIZATION

This section outlines the quality assurance responsibilities of the laboratory staff. An organizational chart and listing of laboratory staff are included in Appendix B.

2.1 QUALITY ASSURANCE RESPONSIBILITIES

The quality assurance responsibilities of the En Chem staff are listed below:

- The Laboratory Manager:
 - Reports directly to the President of En Chem, Inc.
 - Is responsible for the direction of the Quality Assurance Program within the laboratory.
 - Receive reports of quality assurance inspections or audits and promptly take corrective actions in response to any deficiencies.
 - Maintains the current laboratory organization chart.
 - Assure the personnel clearly understand the functions they are to perform in the laboratory.
 - Is responsible for laboratory participation in interlaboratory proficiency programs.
 - Is responsible for directing laboratory certification program.
- The Quality Assurance Officer:
 - Establish and maintain QA and QC systems to accomplish the Quality Assurance objectives.
 - Assess the QC data to insure that analytical systems are operating in a state of statistical control.
 - Conduct data, system and performance audits to monitor completeness and effectiveness of QC systems.
 - Maintain up-to-date Standard Operating Procedures (SOP's), analytical methods and project specific QA plans.
 - Interact with state and federal agencies in matter pertaining to regulations, certifications, methodology, audits and performance evaluation samples.
 - Assure that nonconformance items are documented and resolved.

- Interact with En Chem clients, and regulatory personnel in the preparation and review of Quality Assurance Project Plans.
 - Coordinate the participation of performance evaluation sample programs.
 - Coordinate laboratory participation in certification programs with states and other agencies.
 - Establishes a laboratory quality assurance training program.
 - Reports to the Laboratory Manager on the status of quality control program and audit results.
- The Quality Assurance Coordinator:
 - Conducts laboratory quality assurance audits.
 - Performs statistical analysis on quality control data for all laboratory section.
 - Maintains performance evaluation sample programs and results.
 - Maintains extensive records and archives of quality assurance data.
 - Conduct data reviews on CLP data packages and routine data review.
 - Reports to the quality assurance officer on the status of quality control programs and audit results.
 - Assists in preparation of laboratory SOP's, methods and quality assurance manual
 - Distribution and archiving of laboratory quality assurance documents.
 - Acts as backup to quality assurance officer.
 - Assists in agency visits and external audits of the laboratory.
- The Inorganic and Organic Supervisory staff:
 - Provide technical overview of the inorganic and organic groups.
 - Are responsible for training and continuing compliance of analysts with methods, standard operating procedures, and quality assurance requirements.
 - Serve as technical specialists to adapt methods in areas of new or unique technologies.
 - Review data generated by their staff.
 - Serve as technical specialists and consultant on analytical data generated in the inorganic and organic groups.

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- Define the preventive maintenance and calibration programs for laboratory instrumentation.
- Establish standards for, and maintain, laboratory supplies, chemicals, and standards.
- Review and approve laboratory reports.
- Are responsible for instrument performance, calibration, and preventive maintenance.
- Report out-of-control situations to the Quality Assurance unit by completing nonconformance reports.
- Maintain adequate and appropriate quantities of laboratory supplies.
- The Analysts/Sample Preparation Personnel:
 - Perform methods, data recording and data validation using prescribed methods according to written procedures.
 - Report out-of-control situations to the Quality Assurance unit by completing nonconformance reports.
 - Are responsible for instrument performance, calibration, and preventive maintenance.

3.0 STANDARD LABORATORY PRACTICES

There are many laboratory functions that need to be controlled before and after analysis to produce good quality data. These functions, along with the actual analysis, comprise the daily Quality Assurance Program. It must be recognized that each quality function is, to some extent, dependent on those which preceded it. This means that each quality function must be controlled or specified, and verification of the steps taken must be documented. This section summarizes those quality functions that are discussed in more detail in Sections 4.0 through 13.0.

3.1 MATERIAL PROCUREMENT

The grades of chemicals, solvents, gases, and water will be specified in the Standard Operating Procedure for each method. Likewise, glassware and containers used in the laboratory shall meet or exceed method requirements. All chemicals and materials will be stored in a manner to preserve their integrity and usability until needed.

3.2 LABORATORY SAMPLE HANDLING

Procedures are defined for sample chain of custody from collection through receipt, storage, analysis, and disposal. Proper sample handling throughout the entire laboratory process is essential to producing and delivering data of known quality to our clients.

3.3 INSTRUMENT CALIBRATION AND PREVENTIVE MAINTENANCE

Calibration may be performed using either national standards (for instruments that measure parameters such as mass, time, and temperature) or using chemicals of known composition and concentration. It may

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be performed daily as part of instrument usage or at specified calendar periods, such as quarterly or yearly. See Section No. 6.0 for more information on calibration procedures.

Preventive maintenance involves scheduling regular service for instruments, maintaining an adequate inventory of spare parts, and keeping instrument log books so that the performance of an instrument over time can be assessed. See Section No. 7.0 for details of this program.

3.4 ANALYTICAL PROCEDURES

Analysis shall be performed according to approved En Chem Standard Operating Procedures. USEPA-approved methods are used in the laboratory. See Section No. 9.0 for a listing of Method references. Method performance data will be maintained on file in the laboratory.

3.5 DATA QUALITY CONTROL

Verification of analytical data requires: 1) calculation of quality control data, 2) comparison with acceptance limits for accuracy and precision, and 3) independent validation of quality control results by another analyst.

3.6 CORRECTIVE ACTION

A corrective action program shall be in place in the laboratory that defines responsibilities and actions if conditions are identified that are out of control or do not conform to standard laboratory operations.

3.7 DATA HANDLING AND VALIDATION

The Standard Operating Procedures used to perform the analysis will include the formula for all calculations used to generate the analytical results. All data will be verified by a peer review, then reviewed for completeness and accuracy by the group leader or supervisor. A minimum of 10% of all laboratory reports are reviewed by the QA unit for QA compliance, QC criteria, accuracy, and traceability before the report is issued.

3.8 LABORATORY REPORTS

All reports for data generated by En Chem Laboratory will be issued on En Chem Laboratory letterhead. The format and content of the laboratory report may vary depending on specific project or client needs. Laboratory reports will be approved by the Lab Manager, QA Officer, or other authorized staff.

3.9 RECORDS MANAGEMENT

The En Chem Laboratory records management system shall keep all records secure, complete, and retrievable. All laboratory records, from sample receipt through disposal will be available if requested by a client or an authorized agency or Court.

4.0 MATERIAL PROCUREMENT

The quality of chemicals, reagents, solvents, gases, water, and glassware will be specified so that any effect on analytical results can be considered. Materials purchased by the En Chem Laboratory will meet the requirements listed below, or shall be specified in the analytical method. Supervisors are responsible for checking that all materials received meet these specifications.

4.1 SPECIFICATIONS FOR CHEMICALS, REAGENTS, SOLVENTS, AND GASES

A variety of grades of purity, ranging from technical to ultra-pure grades, are available. The grade of materials to be used will be listed in the method in most cases.

4.1.1 Wet Chemistry

For most of this work, Analytical Reagent (AR) grade or primary grade reagents and solvents are of sufficient purity. The grade of materials to be used will be listed in the method in most cases.

4.1.2 Trace Metals Analysis

Reagents, solvents, and standards will be of high purity. Purchased standards meeting method requirements may be used. Commercial grade fuel and oxidant gases are adequate for atomic absorption spectrophotometry.

The following criteria are required for cryogenic argon and nitrogen:

	<u>Argon</u>	<u>Nitrogen</u>
Purity	99.996%	99.999%
Oxygen	< 5 ppm	< 5 ppm
Hydrogen	—	< 1 ppm
Nitrogen	< 20 ppm	—
Hydrocarbons	—	< 1 ppm
Water	< 4 ppm	< 5 ppm

4.1.3 Trace Organics Analysis

Reagents, solvents, and standards will be pesticide grade (nanograde), and free of interferences for the specific method for which they are being utilized. All solvent lots will be checked before use.

4.1.4 Water

ASTM Type II deionized water (maximum electrical conductivity at 25°C of 1.0 umho/cm, or minimum electrical resistivity at 25°C of 1.0 M ohm/cm) is used for preparing and diluting solutions and for the final rinsing of glassware. The water quality will be measured and recorded in a logbook daily. For trace volatile organic work, water with low organic background is necessary. Commercially available distilled water has been found to be satisfactory for this use.

4.1.5 Compressed Air

Compressed air will be free of oil, water, and dirt. Appropriate filters will be used if the air is produced in the laboratory. Purchased air will be high quality, dry grade (zero grade).

4.1.6 Compressed Gases

The grade of compressed gas will meet the objective of the testing procedure and will meet or exceed the manufacturer's requirements.

4.2 SPECIFICATIONS FOR LABORATORY CONTAINERS

The composition and tolerances of containers used in the laboratory can affect analytical results. Specifications for sample containers are included in an appendix to this manual.

4.2.1 Composition of Containers

Chemically resistant borosilicate glass (e.g., Pyrex, Kimax) is recommended for general use in the laboratory. Plastic polyethylene containers may be suitable for some uses. Disposable glassware is acceptable for many analyses. Inorganics reagents and standard solutions will be stored in borosilicate or polyethylene bottles. Dilute metal standards will be prepared at time of use because they may plate out on the surface of the container. Standard solutions of silica, boron, and alkali metals will be stored in polyethylene bottles.

Organic reagents and standard solutions will be stored in borosilicate bottles.

4.2.2. Specifications for Volumetric Glassware

Volumetric flasks, pipettes, and burets will be Class A unless a less accurate grade is specifically permitted by the method.

4.3 STORAGE OF CHEMICALS, REAGENTS, AND SOLVENTS

Chemicals, reagents, and solvents will be stored in accordance with the manufacturer's recommendations.

Light-sensitive materials must be kept in amber bottles in the dark. Organic reference and reagent standards material are stored at or below 4°C. Standards must be stored separately from samples. When fresh standard solutions are prepared, they should be checked against the old standard or a standard reference material. All reagents will be dated when received and opened. All prepared reagents will be labeled in accordance with approved standard operating procedures.

4.4 CLEANING OF GLASSWARE

Water soluble material can be removed with hot or cold water and rinsed with Type II deionized water.

Detergents, acids, or organic solvents may be required for other substances. All glassware should be rinsed immediately after use to make cleaning easier. SOPs for cleaning glassware will be maintained in the organic and inorganic areas. Glassware will be segregated, and not exchanged amongst wet chemistry, metals, and organic sections

5.0 SAMPLE CONTROL AND PROCESSING

Sample collection, preservation, and storage, before analysis must be performed properly so as not to adversely affect sample integrity. Upon arrival, all samples must be properly logged into the laboratory. The purpose of the sample log-in procedure, which includes assignment of unique sample identification numbers, is to ensure samples can be tracked, data can be stored, and quality control samples can be identified for all analyses occurring in the laboratory.

The following sections detail laboratory procedures related to sample control and processing. Many specific procedures are detailed in laboratory Standard Operating Procedures (SOPs). Any deviations from the procedures presented in this section, or in the SOPs, must be documented in accordance with Section 13 (Corrective Action) of this manual.

5.1 BOTTLE REQUEST, CHAIN-OF-CUSTODY, AND WORK ORDER FORMS

Field personnel are responsible for completion of the bottle request form (Fig. 5-1). The completed request form ensures the proper bottle types and preservatives are made available for the project sampling plan. The completed bottle request form is submitted to the laboratory Receiving group before the sampling event. Field personnel must also complete the sample Chain-of-Custody (COC) Form (Fig. 5-2). This form must accompany samples to the laboratory. This is critical if holding times are to be met. Any unusual requests such as lower detection limits or additional QC, that are specified on the work order, or project QAPP, will take precedence over this QA Manual if they conflict.

5.2 CHAIN-OF-CUSTODY PROCEDURES

It is extremely important to be able to demonstrate that the samples analyzed came from the stated locations and reached the laboratory without alteration. A COC serves as a written record of sample possession and transference. A sample is considered to be in custody if it is in one's possession, is locked or sealed during shipment, or is placed in a secure area limited to authorized personnel. The COC must be signed and dated by everyone who takes possession of the samples.

When samples are shipped by commercial carrier the COC must be sealed in a watertight container and placed in the shipping container. The shipping container must be sealed before delivery to the commercial carrier. The waybill of the carrier serves as an extension of the COC between the field and the laboratory. Upon arrival at the laboratory, the shipping container will be opened in the sample log-in area. The contents are checked against the COC and any discrepancies are noted on the COC. All additions or changes to the COC must be signed and dated at the time that they are made. If the discrepancies cannot be resolved, project personnel will be notified and the samples will be held until the problem is resolved. The laboratory will not be responsible for meeting holding times on these types of problem samples. A Nonconformance Memo will be used to document actions taken to resolve problems with incoming samples (see Section 13, Corrective Action).

5.3 SAMPLE RECEIPT

Samples will be logged in immediately upon receipt during work hours. Samples that arrive during non-business hours will be logged in immediately the next business day.

The following actions are to be performed at sample log-in:

1. Remove samples from the cooler and group together according to sample point.
2. Record the temperature of the temperature blank contained in the cooler. If a temperature blank is not present, record the temperature of the cooler water. As an alternative, a representative (unpreserved) sample may be used. Do not use a sample requiring no headspace for temperature determination. Take care to avoid cross contamination of samples.

Occasionally samples received from nearby sites have not had sufficient time to cool down to 4°C. In those cases, measure the actual temperature, log in the samples, and place them in the refrigerator immediately.

3. Verify cooler contents against container inventory on COC. Record any discrepancy on COC and document accordingly. Sign the COC as received by the laboratory.

Examples of discrepancies include:

Broken, missing, or empty bottles

Samples with low volume

Samples requiring additional preservative

- 4 Analyze the pH of all preserved samples, except those requiring no headspace. Add preservative if not within the required range. Record laboratory pH adjustment on a nonconformance memo.
- 5 Assign sample numbers and enter samples into master log book (Fig. 5-3). Record assigned numbers on the COC and the work order. Place sample identification numbers on sample containers. Take care to associate the proper bottle with the proper sample number using the sample point description on the COC and bottle label.
- 6 Record the sample identification numbers, client information, and other applicable information on a Laboratory Tracking Sheet (LTS) (Fig. 5-4) and store samples in the appropriate location.
- 7 Review the work order for tests that have a short holding time. Notify laboratory supervisors of any tests for which there is a short holding time by placing a notification in the "HOT BOX" for the appropriate group.

The Section Supervisors are responsible for prioritizing work to ensure that holding times and project commitments are met.

- 8 Enter samples into the Laboratory Information Management System (LIMS).

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LIMS entry is checked and verified against the COC and work order submitted with the samples

Nonconformance memos generated during the entry process are also reviewed for resolution and routing requirements

5.4 SAMPLE STORAGE

The primary considerations for sample storage are proper temperature (4°C) and the completion of extraction and analysis within the specified holding times. Sample receiving personnel have the primary responsibility for ensuring that samples are placed in the proper environment.

Sample containers are logged into the proper storage location. Samples requiring refrigeration are logged into the walk-in or remote refrigerators located throughout the laboratory. Samples not requiring refrigeration are placed on shelves in the archive room. The location of sample storage is documented on the LTS sheets for those samples.

To minimize the possibility of contamination all samples for volatile organics or gasoline analysis are placed in leakproof zip-lock bags. This applies to all 40-ml vials and 60-ml jars or any sample to be stored in the Volatile Organics refrigerator. Bottles with the same sample number (i.e. sampled in triplicate) may be stored in the same bag.

5.5 SAMPLE DISPOSAL

Samples may be completely consumed during analysis, returned to the client or sampling location, stored under required environmental conditions if re-analysis is anticipated, or under ambient conditions if re-analysis is not likely, or, disposed of by the laboratory. Samples and extracts shall be disposed of within thirty days after issuance of the final report unless otherwise specified.

En Chem, Inc., is classified as a small-quantity generator by the USEPA

Disposal of all samples, hazardous and nonhazardous, will be performed in accordance with all applicable local, state, and federal environmental regulations. Some nonhazardous wastes may be disposed of in a sanitary sewer as permitted by 40 CFR 261.3 (a),(2),(iv). Hazardous wastes as defined under 40 CFR 261 are stored in several designated locations in the laboratory according to EPA standards.

En Chem, Inc., has an agreement with a licensed hazardous waste shipper; this shipper packs, tests, and ships the hazardous waste quarterly. Hazardous wastes are shipped to licensed waste disposal facilities for disposal.

References	29 CFR 1910.1450
	40 CFR 761
	40 CFR 761.3
	40 CFR 261.3 (a), (2), (iv)
	40 CFR 261.4 (d), (e), (f)
	40 CFR 262
	40 CFR 172.604

Bottle Request Form

EN CHEM FIELD BOTTLE REQUEST FORM

Sample Type

Checked By

- 2) Is there residual chlorine in the water samples? Y N

[illegible]

Rate for sample return

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Chain of Custody Form

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Master Logbook Page

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Fig 5-4
 Laboratory Tracking Sheet

LTS

SHELF	SAMPLE NUMBERS	BOTTLE TYPE	IN	OUT	IN	OUT	IN	OUT	IN	OUT	IN	OUT	IN	OUT	FINAL OUT

Dispose _____	Comments _____
Disp date _____	
Disp by _____	
Storage _____	
Begin date _____	
End date _____	
Disp date _____	
Return _____	Generator: Client In-house
Return date _____	Archive room storage location _____
Return via _____	

6.0 CALIBRATION

Instruments and equipment used to generate data will be calibrated with sufficient frequency, and in such a manner that accuracy and reproducibility of results are consistent with the manufacturer's specifications.

Calibration may be of two types: operational calibration which occurs before instrument use, or periodic calibration which occurs at prescribed intervals. This section describes procedures for maintaining the accuracy of all instruments and measuring equipment that are used for conducting laboratory analysis.

6.1 CALIBRATION REFERENCE STANDARDS

Physical standards (e.g., weights, thermometers) shall be traceable to nationally recognized standards such as the National Institute of Standards and Technology (NIST), which are at least four to ten times as accurate as the equipment requirements. Physical standards will be recalibrated every three years by a certified external agency.

Chemical reference standards will be certified by NIST Standard Reference Materials (SRMs), standards provided by the United States Environmental Protection Agency (USEPA), or vendor-certified materials traceable to these standards. Certificates which accompany standard materials when received in the laboratory will be maintained on file in the laboratory.

6.2 OPERATIONAL CALIBRATION

Operational calibration involves measuring a standard response or preparing a standard calibration curve.

Calibration procedures for a specific laboratory instrument will consist of an initial calibration, initial calibration verification, and continuing calibration verification. Detailed calibration procedures for a specific

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laboratory instrument are documented in En Chem laboratory Standard Operating Procedures (SOP's).

The SOP for each method performed in the laboratory will describe the calibration procedures, their frequency, acceptance criteria, and the conditions that will require recalibration. The analyst is required to perform and document the calibration procedure before sample analysis.

6.2.1 Documentation

Calibrations are documented in the sample logbook for each instrument. The following information will be recorded or referred in the logbook: instrument identification, calibration date, analyst, calibration solutions run, and the samples associated with each calibration.

6.3 PERIODIC CALIBRATION

Periodic calibration is performed for laboratory equipment such as balances and thermometers, which are not calibrated as part of an analytical procedure.

6.3.1 Calibration of Analytical Balances

All balances will be calibrated annually by an external agency using weights traceable to the NIST.

Calibration will be verified daily with reference weights (Class S or better), and the calibration will be documented in the log book.

6.3.2 Calibration of Thermometers

A certified thermometer traceable to NIST is used to calibrate working thermometers. The certified thermometer will be recertified every three years. Working thermometers will be compared with a certified thermometer every twelve months.

Thermometer Calibration: Thermometers will be calibrated against a NIST-traceable reference thermometer by immersing both thermometers in a bath of an expected known temperature such as freezing (0°C) or boiling (100°C) and comparing the readings. If the error is more than 10%, then the thermometer should be discarded and replaced.

Records of periodic calibration will be filed and maintained by the Quality Assurance department.

6.3.3 Monitoring of Ovens, Refrigerators, and Freezers

The temperature of all drying ovens, refrigerators, and freezers will be measured with a working thermometer daily, and the temperature will be recorded in a logbook.

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7.0 PREVENTIVE MAINTENANCE

Preventive maintenance is performed to ensure proper instrument and equipment performance and to minimize the occurrence of instrument and equipment failure during use. Factors considered when scheduling or performing preventive maintenance include: instrument type, equipment and parts that are subject to wear, deterioration or other changes in operational characteristics, spare parts that should be available to minimize downtime, and the frequency that maintenance is required.

Maintenance must be performed when instrument performance begins to deteriorate as made evident by calibration failure, loss of sensitivity, or failure to meet quality control criteria.

All major equipment in the laboratory is covered under manufacturer service contracts. Periodic preventive maintenance is performed by manufacturer service technicians. Daily or routine preventive maintenance is performed by the analyst responsible for the instrument. Group leaders and section supervisors will monitor this activity.

An adequate supply of consumable parts and hardware will be maintained to ensure continued instrument operation. Table 7-1 summarizes routine preventive maintenance performed in the En Chem laboratory.

7.1 DOCUMENTATION OF PREVENTIVE MAINTENANCE

Each instrument will have a maintenance log that is kept by the instrument. All maintenance performed must be documented in the instrument maintenance logbook. This includes maintenance performed by instrument manufacturer, and service technicians, as well as routine maintenance performed by the analyst. The record of maintenance will note any parts replaced as well as observations made.

Table 7-1
 PREVENTIVE MAINTENANCE GUIDANCE

INSTRUMENT	ITEM CHECKED/SERVICED	FREQUENCY
Gas Chromatograph Volatile Organics	Check Hall Furnace Temperature Check Hall Propanol Flow Check PID Lamp Change PID Lamp Clean and Bake Purge Vessels Change Trap on Concentrator Check Carrier Gas Flow Change Carrier Gas Tanks Check Column Flow Check ELCD Solvent Levels Clean Transfer Lines Change Column	Daily As needed Daily As needed Daily Daily Daily Daily When supply < 250psi. Daily Daily As needed As needed
Gas Chromatograph Semivolatile Organics	Change Septum Check Carrier Gas Change Carrier Gas Tanks Check Gas Flow Rates Change in-line Filters Clip Guard Column (one foot) Return ECD for Service * Perform ECD Wipe Test Clean Nitrogen-Phosphorus Detector Clean FID Replace Injection Port Liner Replace Injection Port Seal Check Syringe Change Syringe	Each Run/ As needed Daily When Supply < 250psi. Daily/ As needed Annually/ As needed As needed As needed Semi-Annually As needed As needed Column change/ As needed Column change/ As needed Daily As needed

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Table 7-1
 PREVENTIVE MAINTENANCE GUIDANCE

INSTRUMENT	ITEM CHECKED/SERVICED	FREQUENCY
GC/MS	GC/MS maintenance is the same as GC with the following additions: Check pump oil Change pump oil* Clean Source Verify Pressures	Bi-weekly Semi-Annually As needed Daily
Gel Permeation Chromatograph	Calibrate System Check Solvent Flow and Pressure Repack Column Perform Pump Maintenance	Weekly when in use Daily As needed Every 1500-2000hrs. of use
pH Meter	Electrolyte Changed	Checked daily, changed as needed
Total Organic Carbon Instrument	Change tin moisture trap Change pump tubing Change drier tube	As needed Monthly or as needed Monthly
Total Organic Halide Instrument	Replace titrant in titration cell Change Quartz inlet and outlet tubes Change pyrolysis tube	Daily As needed As needed
Lachat Quickchem IV	Clean each port of the valve Replace pump tubes Clean unions of the valve Replace O-rings Clean fitting of manifolds	Weekly Monthly As needed As needed As needed
Refrigerators and Ovens	Temperature checked and logged daily	Daily
Walk-in Coolers	Temperature checked and logged daily	Daily

Table 7-1
 PREVENTIVE MAINTENANCE GUIDANCE

INSTRUMENT	ITEM CHECKED/SERVED	FREQUENCY
Atomic Absorption Spectrophotometer	Clean furnace windows Change graphite tube Check gases Check level of water coolant reservoir Replace tubing (CVAA) Replace contact rings Clean optics Clean and adjust furnace head Software file clean-up	Daily As needed Daily Daily As needed As needed Performed in service contract Performed in service contract Monthly
Inductively Coupled Plasma Spectrophotometer	Change pump tubing Run BEC (Background Equivalent Check) to check sample introduction system Check level of water coolant reservoir Clean, realign torch Clean nebulizer, replace tips Check waste tubing and container Software file clean-up Change oil in vacuum pump	Daily or as needed Daily Daily As needed As needed Weekly Weekly Yearly
Analytical Balance	Internal Weight Train, Gears, Electronics	Annual Service
Balances	Service representative calibration	Annually
Deionized/Organopure Water	Hardness check Ion exchange bed changed Replace filters	Daily Weekly As needed by serviceman
Vacuum Pumps and Air Compressor	Check performance Lubrication, belts, etc.	Weekly As needed

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8.0 ANALYSIS OF QUALITY CONTROL SAMPLES

Each specific analytical method includes the quality control requirements which are to be performed to ensure that the data produced is of known quality. Quality control samples shall be analyzed as required by the specific method, laboratory SOP, or, project Quality Assurance Project Plan (QAPP).

8.1 QUALITY CONTROL LEVELS

As can be seen from the following paragraphs, there are many types of QC samples which may be applied to different projects at varying frequencies. These may not be reported to the client, may be reported in summary fashion, or may be reported in detail with all raw data provided. The level of QC must be specified at the time that the samples are submitted. Project specific requirements should be discussed with the laboratory before the samples are taken.

8.2 LABORATORY QUALITY CONTROL SAMPLES

En Chem standard practice is listed in Table 8-1.

8.2.1 Laboratory Control Sample (Blank-Spike Control)

A laboratory control sample (LCS) consists of a control matrix which has been spiked with the analytes of interest or compounds representative of those analytes. For the organic section, the method blanks also serve as the LCS due to the surrogates spiked into the blank. Certified reference materials may also be used as an LCS.

Laboratory control samples must be prepared and analyzed with each group of samples to verify that the analytical process is operating in control. LCS recoveries are then compared to laboratory established control limits. All samples that were prepared with an LCS which falls outside of the established control limits must be repeated.

8.2.4 Method Blank Analyses

A method blank is a volume of deionized water or sample of purified soil/sediment that is carried through the entire analytical procedure to verify that interferences caused by contaminants in the solvents, reagents, glassware, etc. are known and minimized. A method blank shall be analyzed with each group of samples. Ideally, a method blank shall be below the EQL for the compounds of interest. If the method blank is greater than the EQL, all samples prepared with that blank must be repeated. For volatile organics analysis, common laboratory solvents (methylene chloride, acetone, and 2-butanone) are permissible to five times the EQL. In metals analysis a value greater than the EQL is acceptable in the method blank if the analyte of interest is found to be greater than 10 times the EQL in the samples associated with the blank. Otherwise samples must be re-digested. For methods with no preparation step, the initial calibration blank shall also serve as the method blank.

8.2.8 Laboratory Matrix Spike Analyses

To evaluate the effect of the sample matrix upon analytical methodology, a separate aliquot sample is spiked with the analyte of interest and analyzed with the sample. If the percent recovery falls outside established limits, the sample data shall be carefully evaluated to determine what remedial action is required.

8.2.9 Laboratory Matrix Spike Duplicate Analyses

A separate aliquot sample is spiked with the analyte(s) of interest and analyzed with the associated sample and sample matrix spike. The Relative Percent Difference (RPD) will be calculated between the MS and MSD. If the RPD is outside of the established control limits, the sample data shall be carefully evaluated to determine what remedial action is required. For some analyses where precision can be

determined from duplicate sample analysis, the sample duplicate data will be used to determine precision. In this case the RPD will be calculated as above and compared with the control limits.

8.2.5 Duplicate Sample Analyses

Duplicate analysis may be used to calculate the precision (relative percent difference) of an analysis in cases where the levels of analyte is sufficiently above the EQL, or a spike of the analyte is not possible (i.e. TSS). Frequency of duplicate analyses may be either one sample per similar matrix group of 10 or one sample per group of 20, depending on choice of methodology. If the RPD is outside of the established control limits, the sample data shall be carefully evaluated to determine what remedial action is required.

8.2.1 Trip Blank Analyses

Volatile organics samples are susceptible to contamination by diffusion of organic contaminants through the Teflon-faced silicon rubber septum of the sample vial. Trip blanks are prepared by filling two 40-mL VOA vials with organic free water, shall be shipped with the field kit, and follow the sample bottles through the field collection and shipment to the laboratory. Trip blanks are analyzed and reported in the same manner as samples.

8.2.7 Surrogate Standard Analyses

A surrogate is an organic compound which is similar to the target analytes in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples. For volatile and extractable organics, all samples and blanks shall be fortified with surrogate spiking compounds before purging or extraction to monitor sample preparation and analysis. If the percent recovery falls outside established limits, the sample data shall be carefully evaluated to determine what remedial action is required. Reextraction, reanalysis, or a sample narrative may be necessary.

8.2.8 Serial Dilution

For ICP metals analysis, a serial dilution is performed on each matrix spike QC sample. The Matrix spike is diluted by a factor of 5, and the result must agree within 10% of the original matrix spike recovery. Any results that are greater than 10% will require a flag on the sample result indicating an estimated concentration due to a chemical or physical interference.

8.2.9 Dilution Test and Recovery Test

For GFAA metals analysis, a dilution test is performed for each matrix type in each digestion group. To perform the dilution test the concentration of the analyte of interest must be at least 20 times the EQL. A 1:5 dilution is analyzed and the value must be within 10% difference of the original determination to indicate the absence of interferences. If the difference is greater than 10% or, the samples in the digestion group below the EQL, a recovery test is performed. One sample is spiked with a known amount of the analyte of interest and analyzed. The % recovery must be within the range of 85-115. If the recoveries are outside of that range subsequent dilutions are performed. If recoveries remain outside of the acceptable range then all samples in the digestion group are analyzed by the method of standard additions.

8.2.10 Blank Spike Analyses (For Organics analysis)

A blank spike is a volume of deionized water or a sample of purified soil/sediment that is spiked with the analytes of interest and carried through the entire analytical procedure to demonstrate that the laboratory techniques for this method are in control. This sample is recommended in conjunction with matrix spike/matrix spike duplicate samples where severe matrix interferences are anticipated. If the matrix spike/matrix spike duplicate shows poor recoveries while the blank spike sample is acceptable, this is

strong evidence that the method has been performed correctly by the laboratory but that matrix interferences have affected the results of these samples.

8.3 Additional Quality Control samples

8.3.1 Field Blank Analyses

A field blank is a volume of deionized water, or purified soil, that is placed into sample containers at the site by the sample takers and is shipped with the field samples. field blanks are analyzed and reported in the same manner as samples.

8.3.3 Rinsate Blank Analyses

A rinsate blank is a volume of deionized water that is used to rinse a sampling equipment. It is collected after decontamination and prior to sampling. This blank is useful in documenting adequate decontamination of sampling equipment. Rinsate blanks are analyzed and reported in the same manner as actual samples.

8.4 Performance Evaluation Samples

The laboratory participates in the following programs.

- US-EPA Water Pollution
- US-EPA Water Supply
- Wisconsin State Lab of Hygiene
- Analytical Standards Inc. -Double blind
- NIOSH Environmental Lead Proficiency Analytical Testing Program

Table 8-1
 Quality Control Sample

Type	Purpose of Sample	Frequency	Applicability	
			Inorganic	Organic
Laboratory Control Sample	Control matrix spiked with analyte (s) of interest or representative compounds (surrogates) Analyzed to demonstrate method performance and control % recovery is charted	With each group of samples prepared	X	X
Method Blank	To verify that interferences from solvents, reagents, glassware etc. are known and minimized	With each group of samples, as specified in analytical method	X	X
Matrix Spike	A known concentration of a specific parameter is added to an aliquot of a sample with the matrix of interest Percent recovery is determined for accuracy	SW-486 1 out of 20 samples or 1 per batch EPA /600 Series 1 per 10 samples	X	X
Matrix Spike Duplicate	Percent recovery is determined and compared against matrix spiked sample Relative percent difference is determined for precision	SW-846 1 out of 20 samples or 1 per batch	X	X
Duplicate	An aliquot of a sample known to Analyst Calculate relative percent difference (RPD)	SW-846 At least 1 out of 20 where MS/MSD is not possible such as Total Solids determination EPA/600 Series 1 per 10 samples	X	

Table 8-1 cont
 Quality Control Sample

Type	Purpose of Sample	Frequency	Applicability	
			Inorganic	Organic
Trip Blank	Used to verify that contamination of soil / water VOA samples has not occurred due to sample container or during sampling or shipping	Sent with each shipment cooler of VOA vials, or as specified in Project Workplan		X
Surrogate	for volatile and extractable organics, to monitor sample preparation and analysis efficiency	All standards, method blanks, and samples		X
Serial Dilution	Used to verify the absence of matrix interferences for ICP analysis dilution is performed on sample matrix spike	Performed on each matrix spike	X	
Dilution Test and Recovery Test	Used to verify the absence of matrix interferences for metals analyzed by GFAA	One per matrix type for each digestion group	X	
Blank Matrix Spike	Control matrix spiked with matrix spike solution. Used to demonstrate proper execution of method when working with difficult sample matrix.	In conjunction with sample matrix spike and matrix spike duplicate on as needed basis.		X

210 0.10

9.0 ANALYTICAL PROCEDURES

9.1 ANALYTICAL METHODS

Whenever possible, the En Chem Laboratory shall use recognized analytical methods from the USEPA, APHA, ASTM, NIOSH or Standard Methods for the Examination of Water and Wastewater (see Table 9-1).

9.2 DETECTION LIMITS

All methods have detection limits below which an analyte cannot be measured accurately. A detection limit quantity usually shall be reported as a less than value (<). This does not mean that an analyte is not present but only that, if it is present, it is at levels below the detection limit. For results produced by USEPA CLP methods, values below the detection limit may be reported inside brackets [] as "estimated concentrations." It is important to remember that detection limits are highly matrix dependent and the detection limits listed in the method are provided for guidance and may not always be achievable.

9.2.1 Method Detection Limits

Detection limits or instrument detection limits are determined using the following procedure:

Seven to ten samples/replicates which are spiked and analyzed at a concentration that is 1 to 5 times the assumed detection limits. Each analysis is assumed to be a different sample analysis (rinses, proper calibration, injections, etc.). The results are averaged and the standard deviation (n-1) is performed. The following equation is then used:

$$\text{Detection Limit} = t_{(n-1, 1-\alpha=0.99)} (S)$$

Where:

$t_{(n-1, 1-\alpha=0.99)}$ = the students' t value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom.

S = standard deviation of the replicate analysis.

9.2.2 Inorganics

Wet chemical techniques for nutrient, demand, and mineral constituents are relatively free from interferences in the analysis of aqueous samples. Analyses of these constituents in solid phase samples are not routinely performed. High concentrations of any of these analytes require dilution of the sample, with corresponding changes to detection limits.

9.3 VARIANCE FROM STATED ANALYTICAL METHODS

Analyses shall be performed in accordance with approved methods unless specific project requirements or needs dictate adoption of an alternate method or modification of the cited methods. The use of alternate or modified methods must be approved by the laboratory Quality Assurance Officer and also any applicable authority (i.e. Project Manager, WDNR, NEESA) prior to implementation by the laboratory.

Table 9-1
Method References

1. Federal Register, Volume 44, No. 223, December 3, 1979, 40 CFR Part 136, pp. 69464 to 69575.
2. "Guidelines Establishing Test Procedures for the Analysis of Pollutants under the Clean Water Act," CFR Part 136, October 26, 1984.
3. "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol, and Pesticides in Water and Wastewater," Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio, September 1978.
4. "The Analysis of Aromatic Chemicals in Water by the Purge and Trap Method," Method 503.1, U.S. Environmental Protection Agency, Physical and Chemical Methods Branch, Cincinnati, Ohio, May 1980.
5. "Methods for Analysis of Inorganic Substances in Water and Eluvial Sediments," U.S. Department of the Interior, U.S. Geological Survey, Open-File Report 85-495, 1986, and USGS TWRI, Book 5 (1972 and 1979).
6. "Official Methods of Analysis of the Association of Official Analytical Chemists," methods manual, 17th ed.
7. Standard Methods for the Examination of Water and Wastewater American Public Health Association.
8. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846, USEPA, (3rd Edition).
9. "Methods for Chemical Analysis of Water and Wastes," EPA 600/4-79-020, U.S. Environmental Protection Agency, March 1979.
10. "Annual Book of ASTM Standards, Sections 11.01 and 11.02, Water and Environmental Technology," American Society for Testing and Materials.
11. "American National Standard on Photographic Processing Effluents," American National Standards Institute (ANSI).
12. The Determination of Polychlorinated Biphenyls in Transformer Fluid and Waste Oils," Physical and Chemical Methods Branch, Environmental Monitoring and Support Laboratory, USEPA, Cincinnati, Ohio, April 1981.
13. "Interim Methods for the Sampling and Analysis of Priority Pollutants in Sediments and Fish Tissue," Physical and Chemical Methods Branch, Environmental Monitoring and Support Laboratory, USEPA, Cincinnati, Ohio, (April 1981).

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Table 9-1
Method References

14. Manual of Analytical Methods for Analysis of Pesticides in Human and Environmental Samples. USEPA, EPA 600/8-80-038 (June).
15. USEPA Contract Laboratory Program Statements of Work (CLP-SOW) OLM01.8 and ILM02.1

10.0 DATA VERIFICATION

Data verification requires: 1) the review of quality control samples for accuracy, precision and completeness and 2) the review of sample data to ensure that the numerical computations are correct and properly reported.

10.1 PROCESSING OF QUALITY CONTROL DATA

10.1.1 Procedures Used to Assess Data Precision and Accuracy

Review of quality control data shall address the following criteria:

- Reagent Blank Evaluation - If high blank values are observed (usually 2 to 5 times the detection limit), laboratory glassware and reagents shall be checked for contamination.
- Field Trip Evaluation - If high values are found, the procedures for sample collection, shipment and laboratory analysis shall be reviewed. If both the reagent/method blank and the trip blank contain significant contamination, the source is probably within the laboratory. High field blank readings may be due to contaminated sample bottles or cross-contamination due to poorly sealed containers.
- Calibration Standard Evaluation - The daily calibration curve shall be evaluated to determine linearity through its full range, and that sample values are within the range defined by the low and high standards. If the curve is not linear ($r \geq 0.995$), the calibration curve shall be rerun. In some cases, sample values must be corrected for nonlinearity by deriving sample concentrations from a graph or by using an appropriate algorithm to fit a nonlinear curve to the standards.

- Duplicate Sample Evaluation - The duplicate results shall be used to calculate the precision for the sample matrix as defined by the relative percent difference (RPD). If the precision value exceeds the control limit, the reason for the nonconformance shall be determined and documented. Corrective action may include re-analysis.
- Matrix and Blank Spike Evaluations - The observed recovery of the matrix spike versus the theoretical spike recovery shall be used to calculate accuracy as defined by the percent recovery. If the accuracy value exceeds the control limit, the reason for nonconformance shall be determined and documented. Corrective actions may include re-analysis for the parameter in question. If interferences are present in the samples spiked, a blank spike may be used to demonstrate that the laboratory technique is in control.
- Check Standard Evaluation - The results of check standard analysis shall be compared with the original calibration curve, and the relative percent difference of the check standard shall be calculated to determine if the calibration system is in control. If correction is required, the check standard shall be re-analyzed to demonstrate that the corrective action has been successful.
- Surrogate Standard Evaluation - The results of surrogate standard determinations shall be compared with the true values spiked into the sample matrix prior to extraction and analysis and the percent recoveries of the surrogate standards shall be determined. Percent recoveries attained shall be in accordance with current EPA recommendations/requirements or laboratory-generated control limits (VOA, BNA, pesticides).

- Analysis Matrix Spikes Evaluation - Suppression or enhancement of instrument signal levels is demonstrated when recoveries are lowered or raised, respectively. Matrix-suppressing agents may be added to the sample or the extract to reduce such effects in routine sample analysis when the evaluation indicates samples are affected in this manner. One to three levels of analysis matrix spike concentrations shall be used to determine the unaffected concentration level native to the sample. The observed recovery versus the theoretical recovery shall be used to calculate the accuracy as defined by the percent recovery. If the accuracy value exceeds the control limits, the reason for the conformance shall be determined. Corrective action may include re-analysis (metals).

10.1.2 Control Charts

Control charts for precision and accuracy shall be established for all major organic and inorganic parameters. A minimum of 20 data points shall be used to establish control limits. Warning limits of two standard deviations and control limits of three standard deviations shall be used in most cases. If control limits become too narrow, the laboratory may choose to adopt wider limits based on regulatory requirements.

10.1.2.1 Calculation of Precision

The precision or relative percent difference (RPD) is defined as the difference (range) of each replicate set, divided by the average value (mean) of the replicate set, times 100. For replicate results D_1 and D_2 , the RPD shall be calculated:

$$\text{RPD \%} = \frac{[D_1 - D_2]}{(D_1 + D_2)} \times 100$$

When RPD is obtained for at least 20 replicate pairs, the average RPD and the standard deviation shall be calculated using

$$\bar{m} = \frac{\sum_{i=1}^n m_i}{n} \quad S_m = \sqrt{\frac{\sum_{i=1}^n (m_i - \bar{m})^2}{n - 1}}$$

Where:

- m = The RPD of a replicate pair.
- \bar{m} = The average of the Relative Percent Difference determinations.
- S_m = The standard deviation of the data set of RPD determinations.
- n = The number of RPD determinations.

When constructing a control chart for a specific parameter, the Warning and Control Limits shall be calculated from the following:

$$\begin{aligned} \text{Upper Control Limit} &= \bar{m} + 3 S_m \\ \text{Upper Warning Limit} &= \bar{m} + 2 S_m \end{aligned}$$

10.1.2.2 Calculation of Accuracy

The accuracy or percent recovery (%R) is defined as the observed concentration of the spike, times 100.

$$\%R = \frac{O_i - O_s}{T_i} \times 100$$

Where:

- %R = The Percent Recovery
- O_i = The Observed Spiked Sample Concentration
- O_s = The Sample Concentration
- T_i = The True Concentration of the Spike

The true concentration shall be calculated:

$$T_i = \frac{\text{Spike Concentration [c] (mg/L) x Volume of Spike (in mL)}}{\text{Volume of Sample [in mL] + Volume of Spike [in mL]}}$$

When the Percent Recovery is obtained for at least twenty spiked samples, the mean percent recovery and the standard deviation shall be calculated using the formula:

$$\overline{\%R} = \frac{\sum_{i=1}^n \%R_i}{n}$$

and

$$S_R = \sqrt{\frac{\sum_{i=1}^n (R_i - \overline{\%R})^2}{n-1}}$$

Where:

- $\overline{\%R}$ = The mean percent recovery
- $\%R_i$ = The percent recovery of a single spiked sample
- n = The number of results
- S_R = The standard deviation of the data set of percent recovery determinations.

The warning and Control Limits shall be calculated from the following equations:

- Upper Control Limit = $\%R + 3 \overline{S_R}$
- Lower Control Limit = $\%R - 3 \overline{S_R}$
- Upper Warning Limit = $\%R + 2 \overline{S_R}$
- Lower Warning Limit = $\%R - 2 \overline{S_R}$

10.2 **DATA VALIDATION** (See Figure 10-1)

Data shall be generated by trained analysts (see Section 16.0) using approved methods and in-control instruments. Data validation shall include sample identification, calculation errors, and transmittal or transcription errors. The final report shall be reviewed and signed by the Laboratory Director or his designee.

10.2.1 Data Processing

Data may be manually computed, input into a computer for processing or calculation, or directly acquired from a computer.

If data are manually processed by the analyst, all steps in the computation shall be provided including equations used and the source of input parameters such as response factors, dilution factors, and calibration constants. These shall be performed on the data sheet or on an En Chem Computation Sheet which shall be initialed and dated by the analyst and attached to the data sheets.

For data entered and processed in a computer, the analyst shall indicate on a copy of the input the sample(s) or project number, sign and date the copy, and attach it to the data sheets.

For data acquired directly from the computer, the analyst shall verify that all parameters (project/sample numbers, response factors, units, detection limits, etc.) are correct. The analyst shall sign and date the output.

10.2.2 Review of Data Processing

One hundred (100) percent of all data shall be checked by a second analyst.

The independent analyst shall check for correct interpretation of charts, for proper equations and calculations, and for correct data input. All entries and calculations that are reviewed shall be indicated with a checkmark or highlighted in yellow or blue ink. The checker shall initial and date in ink all pages of the data package (except for printouts such as chromatograms).

If the checker disagrees with a number, the checker shall mark through the number with a single line, place the revised number above it, and initial the change. Any changes shall be back-checked by the data originator so that any differences may be quickly resolved.

In the same manner, at least 20 percent of all computer input entries shall be checked and agreement indicated by a checkmark or highlighting. Errors shall be marked through with a single line, the correct figure listed above, and the data reprocessed using the corrected input.

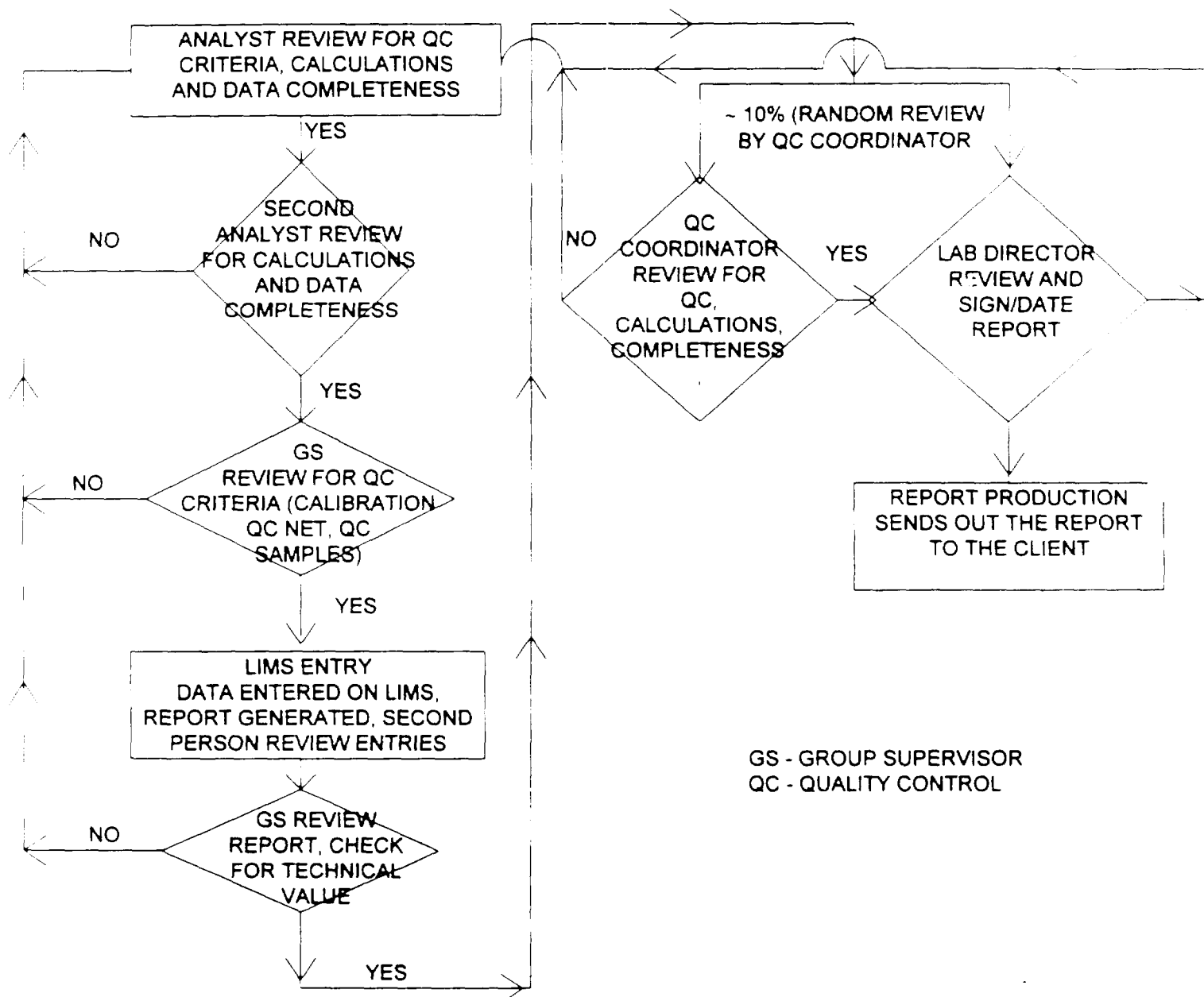
All data entered into the Laboratory Information Management System by administrative staff shall be checked for input errors by a second employee.

10.2.3 Review of Laboratory Reports

All laboratory reports shall be reviewed by the Group Supervisor who shall initial and date a lab report review routing slip. Then, the laboratory report shall be reviewed and signed by the Laboratory Manager or his designee.

10.3 VERIFICATION OF SOFTWARE

Computer software shall be verified by running sample problems that test all the options of the software and by comparing the values to hand calculations. Software shall be verified on an annual basis or whenever it is modified. The analyst shall document this procedure by signing and dating both the computer output and the hand calculations.



11.0 DATA REPORTS

The format and content of the laboratory data report will vary depending on project or client needs, contract and regulatory requirements, and the need for method numbers or explanatory text.

Each page shall list client, project number (if applicable), field and laboratory identification, and sample date (if known). Data shall be presented in a tabular format whenever possible.

Data listed on the report shall include parameters analyzed, reported values, regulatory limits (if applicable), and units of measurement. Detection limits shall be indicated by a "less than" sign (<) or appropriate qualifiers. If necessary, case narrative text shall be included in the report or in a separate letter of transmittal. All reports shall be signed by the Laboratory Manager or his designee. Any analytical results communicated verbally shall be considered preliminary until data are sent in hard copy. A Verbal Results Log shall be maintained in Client Services to record all verbal results given to laboratory clients.

12.0 RECORD MANAGEMENT

Laboratory records fall into two major categories:

- Documents which reflect overall laboratory operation such as instrument log books and control charts.
- Documents which are specific to a group of samples such as chain of custody and raw analytical data.

A good records management system keeps records secure, complete and retrievable. All laboratory records from time of sample receipt through data reporting and sample disposal shall be available if requested by clients or an authorized regulatory agency or court.

12.1 GENERAL LABORATORY OPERATIONS RECORDS

The following records shall be maintained by the laboratory:

- Master Sample Log - A chronological record of all samples entering the laboratory shall be maintained in the sample log-in area.
- Instrument and Calibration Maintenance Logs - A separate log shall be maintained for each instrument listing all maintenance and calibration performed in-house or by outside groups. These logs shall be maintained in the laboratory during use and then archived in the main office.
- Performance Evaluation Records - A record shall be maintained in the Main Office of all laboratory participation in any performance evaluation program (DNR, USEPA, etc.). Copies shall also be kept in the laboratory, and by the laboratory Quality Control Coordinator.
- Certification Program Records - Records shall be maintained in the Main Office of all correspondence, analytical data, agency results and certification of performance from all certification programs.
- Control Charts - Charts shall be filed chronologically for each parameter. Current charts shall be maintained in the laboratory and old charts archived for three years.
- Purchased Material Certificates - Information which verifies that purchased materials meet the requirements of the laboratory shall be maintained in the laboratory.

- Audit Records - Formal audit reports of internal and external performance audits shall be filed by the Quality Assurance Officer.
- Computer Software Verification - Separate record of the data used to verify each software package shall be maintained in the laboratory.
- Training Records - Resumes, external training, and in-house training records shall be maintained alphabetically by name of employee in the Group Supervisor's office.
- Master Nonconformance Record - A copy of all nonconformance reports shall be maintained.
- Instrument Run Log - A list of samples run on each instrument in the organic area shall be maintained in the organic area.
- Internal Chain-of-Custody Records - All records tracing a sample through the laboratory shall be maintained.
- Standard Operating Procedures - A file of current and historical laboratory SOPs with issue dates shall be maintained in the Main Office. SOPs shall be signed and dated by the Quality Assurance Officer and Laboratory Manager.
- Methods - A complete collection of all analytical methods used in the laboratory shall be maintained in both the laboratory and in the Main Office. Methods shall be signed and dated by the Group Supervisors, the Quality Assurance Officer, and the Laboratory Manager.
- Subcontractor Records - Audit reports and results of any QC samples submitted to subcontractors shall be maintained on file with the Quality Assurance Officer.

12.2 PROJECT/SAMPLE RECORDS

Separate record packages shall be maintained for each project and filed by project number.

12.2.1 En Chem Client Files

A file by project number shall be maintained in the Main Office of the laboratory for samples submitted by En Chem employees. The following records shall be maintained:

- Chain-of-Custody Form
- Request for Analysis Form
- Laboratory Data Reports
- Any Correspondence

- Sample Record Form
- Sample Acknowledgment Form
- Any Telephone Messages
- Any Subcontractor Reports

Bench sheets shall be maintained in a separate file by test method and/or by project number.

12.2.2 Laboratory Only Client Files

A file by project number shall be maintained in the Main Office of the laboratory for samples submitted by laboratory only clients. The following records shall be maintained:

- Chain-of-Custody Form
- Request for Analysis Form
- Purchase Order
- Laboratory Data Reports
- Any Correspondence
- Any Telephone Messages
- Sample Record Form
- Any Subcontractor Reports
- Letter of Transmittal
- Invoice

12.2.3 Laboratory Area Files

Files by project number shall be maintained in the laboratory area and shall contain the following information:

- Copy of Chain-of-Custody
- Copy of Request for Analysis Form
- Copy of Extraction Sheets
- Copy of Bench Sheets
- Copy of Work Sheets
- Raw Analytical Data Including Chromatograms

12.3 RECORD CONTROL

The Laboratory Manager shall appoint an individual who shall be responsible for the records management system including initiating new project files, adding records to existing files, and assisting laboratory personnel in withdrawing and returning records.

To maintaining control of records, a sign-out sheet shall be maintained for each of the files (Main Office, Organic, Inorganic) indicating project number, date borrowed, name of borrower, and date of return.

12.4 RECORD RETENTION

Laboratory records shall normally be maintained for seven years after analysis. If a specific contractual requirement, or government regulation, requires that records be maintained for a longer period of time, the project file shall be marked with the required retention period.

12.5 SAMPLE STORAGE

Analytical samples shall be stored for at least thirty days after submittal of the laboratory report before disposal or return to the client.

13.0 NONCONFORMANCES AND CORRECTIVE ACTION

Nonconformances may include the following:

- Instrument failures/problems.
- Incomplete/missing sample documentation.
- Unacceptable sample condition.
- Exceeding sample holding times.
- Improper sample storage.
- Incorrect sample preparation.
- Wrong analysis method/procedure.
- QC data (blank, spike, duplicate, surrogates, etc.) outside acceptance limits.
- Calibration requirements not met.
- Data recording, transcription or validation errors.
- Any other situation that might affect data quality.

In all such cases, a nonconformance memorandum shall be initiated giving a description of the problem, the corrective action taken, the name of the individual reporting the problem, the date discovered, and the samples affected. This memo shall be initiated by the Group Supervisor and filed in the Master Nonconformance Record File. Examples of possible memos are shown in Figures 13.1, 13.2, 13.3, and 13.4.

Corrective action may include the following:

- Recalibrating instruments.
- Re-analyzing samples.
- Instrument repairs.
- Additional training of laboratory personnel.
- Using different lots/solvents to correct for high blank values.
- Notifying clients of missing paperwork, broken containers or insufficient samples.

13.1 RESPONSIBILITIES

All employees shall be responsible for reporting any nonconformance that they observe/identify and for signing the nonconformance memo. Each Group Supervisor shall be responsible for correcting problems that affect data quality and for stopping work when an out-of-control situation is found.

NONCONFORMANCE MEMO

Organic Laboratory

Date: _____
ANALYST: _____

Project Name: _____

Project No: _____

LIMS Test ID: _____

All Sample Number affected: _____

NONCONFORMANCE (related to: _____ Matrix, _____ Prep, _____ Instrument, _____ Other):

CORRECTIVE ACTION(S) TAKEN:

REVIEWED BY:	<u>Initials</u>	<u>Date</u>	<u>Check if</u> <u>Corrected</u>
Group Supervisor	_____	_____	_____
QA Coordinator	_____	_____	_____

2 0 0 0

NONCONFORMANCE MEMO

Inorganic Laboratory

DATE: _____

ANALYST: _____

Project Name: _____ Sample No(s): _____

Project No: _____ LIMS Test ID: _____

NONCONFORMANCE: [Check applicable and describe]:

Matrix related: ☐ Spike ☐ Duplicate ☐ Other

Prep related: ☐ Spike ☐ Duplicate ☐ LCS ☐ Prep Blank ☐ Other

Instrument related: ☐ ICV/CCV ☐ ICB/CCB ☐ % CV ☐ Other

Other:

CORRECTIVE ACTION(S) TAKEN: (Describe):

☐ (Check if report flag needed)

REVIEWED BY:	<u>Initials</u>	<u>Date</u>	<u>Check if</u> <u>Corrected</u>
Group Supervisor	_____	_____	_____
QA Coordinator	_____	_____	_____

SAMPLE ENTRY NONCONFORMANCE MEMO

PROJECT NAME:		PROJECT NUMBER:		INITIATOR & DATE:	
PROBLEM	Bottle Type & Preservation	Lims Number	Station ID	Comments/ Corrective Action	Completed Date/Initials
No ice					
Broken Bottle/ Cracked Cap					
Hold Time					
headspace					
Labeling					
Volume					
Preservation					
Container					
Other					

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14.0 QUALITY ASSURANCE/QUALITY CONTROL AUDITS

Audits of the En Chem Laboratory are conducted for several reasons:

- To identify potential or actual deficiencies so that the problems may be corrected.
- To assure that En Chem procedures and methods are being followed.
- To determine that records are properly filled out and filed.
- To assure that regulatory requirements are met.
- To establish that quality assurance objectives are met.
- Audits are not conducted to assign blame.

14.1 LABORATORY AUDITS

On a quarterly basis, the Quality Assurance department will conduct an in depth audit of specific areas of the laboratory. The areas audited each quarter will be rotated, so that after a period of one year all areas have been audited. The following areas will be audited:

- Sample entry
- Inorganic, Wet Chemistry
- Inorganic, Metals
- Volatile organics
- Semi Volatile organics
- Reporting

The audits shall include the following items:

- Sample maintenance
 - Are stated temperatures for sample storage provided?
 - Are samples processed and tested within prescribed holding times?

- Are samples properly logged in?
- Calibration
 - Are calibrations performed as required?
 - Are they properly documented in instrument log books, or as part of project data if required?
 - Do calibration results indicate a trend in instrument performance?
- Preventive maintenance
 - Are adequate spare parts available?
 - Do specific instruments have repeated maintenance problems?
 - Is preventive maintenance performed and properly documented?
- Receipt and storage of standards, chemicals, and gases
 - Are all reagents, chemicals, and gases purchased for use in the laboratory of adequate grade for the intended use?
 - Are certifications of material compositions provided when required?
 - Are materials adequately stored to prevent degradation?
 - Are materials kept beyond stated shelf life?
 - Are internal standards properly prepared and stored?
 - Are internal standards kept beyond stated shelf life?
- Analytical Methods
 - Are the methods used appropriate for project requirements?
 - Are alternate methods approved for use?
- Data Verification
 - Are data processed and validated as prescribed?
- Records Management

- Are the records of analyses complete and properly identified?
- Are documents submitted to the record system in a timely manner and are they properly maintained?

The laboratory audit shall consist of a general audit and a specific method/procedure audit.

- The general audit shall be an overview of the area being audited for compliance with the Quality Assurance Manual, and adherence to applicable standard operating procedures.
- A specific method/procedure audit shall be a detailed in-depth review of an actual method or procedure. This may include sample receipt, standard/reagent/solution preparation, sample preparation/extraction, sample analysis or data verification.

During the general and/or specific audits, the Quality Assurance Coordinator will document findings using applicable forms. Any problems, observations, and findings which are identified by the Quality Assurance Coordinator shall be discussed with the Group Supervisors.

A written report summarizing the findings of the audit shall be sent to the Group Supervisors who will be responsible for corrective action. A written response including supervisors comments, and corrective actions will be returned to the Quality Assurance officer within a reasonable time. The Quality Assurance department will verify that the corrective actions are appropriate and have been implemented in the laboratory.

A copy of the audit report along with responses shall be routed to the Laboratory Director for review.

Deficiencies reported as a result of participation in round-robin studies or outside audit shall be handled in the same manner.

15.0 QUALITY REPORTS TO MANAGEMENT

On a monthly basis, the Quality Assurance Officer will prepare a quality assurance report to be submitted to the laboratory Director. The report will contain information on the following:

- Corrective Action Program Summary
 - Report on corrective actions implemented since the last report.
 - emphasis given to ongoing or recurring problems.
- Laboratory Audit Results
 - Internal and External Audits.
- Laboratory performance on P.E. Samples.
- Comments from Data Review and Validation.
- Summary of QA/QC programs, training, and other miscellaneous accomplishments.

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16.0 TRAINING

All activities in the En Chem Laboratory shall be accomplished by personnel qualified on the basis of education, experience and training.

16.1 JOB DESCRIPTIONS

All positions affecting data quality shall have written job descriptions which include the minimum qualifications for education, experience, knowledge and skills. During the semi-annual performance review, the laboratory supervisory staff shall compare each analyst's performance with the appropriate job description.

16.2 PROFESSIONAL STAFF, TRAINING AND QUALIFICATIONS

Qualifications of all professional personnel shall be documented by resumes which include academic credentials, employment history, and experience. An analyst hired to perform sample preparation procedures or analytical procedures shall receive direct instruction from a professional staff member on topics such as sample log-in, sample/glassware preparation, use of instrumentation, methods, quality assurance, data handling and safety. When the Group Supervisor feels confident in the skills of the analyst, he shall write a memo detailing the training of the analyst and any qualifying tests that were conducted (analysis of known samples, duplicates, etc.). Technicians and support personnel performing a technical function shall be qualified through experience and this shall be indicated in the resumes and training files. They shall be supervised by experienced personnel until, in the opinion of the Group Supervisor, they are capable of independently performing their duty. The Group Supervisor shall write a memo describing the training that the technician has received, and listing the tests and procedures for which they have qualified.

16.3 QUALIFICATION AND TRAINING RECORDS:

A file shall be maintained containing the qualifications and training records of each laboratory employee.

This file shall contain resumes, lists of all technical training courses completed, certificates of training, and all memos detailing training at En Chem.

APPENDIX B

EN CHEM SOP FOR

TOXAPHENE SOIL EXTRACTION

AND ANALYSIS

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Standard Operating Procedure

TITLE: Sulfuric Acid Cleanup

DEPARTMENT: Semivolatile Organic Extractions

APPLICATION: A variety of interfering organic compounds in the polychlorinated biphenyl analysis are destroyed by the addition of concentrated sulfuric acid to the sample extract.

REFERENCES: Test Methods for Evaluating Solid Wastes
SW846 method 3665A (3rd Ed., Rev. 1, July 1992)

PROCEDURE SUMMARY:

The sample extract undergoes cleanup by the addition of concentrated sulfuric acid. The mixture is shaken and the extract is removed from the cleanup reagent.

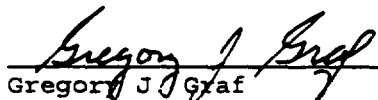
REVIEWED BY:



Daniel M. Rude
Organics Group Leader
Organic Laboratory

8-30-96

Date



Gregory J. Graf
Quality Assurance Officer

9-3-96

Date

APPROVED BY:



Eric L. Thomas
Laboratory Manager

9/6/96

Date

QUALITY CONTROL:

- When this cleanup procedure is used, all sample, blank, and matrix spike extracts undergo this process.

INTERFERENCES:

Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the total ion current profiles (TICPs). All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerable from source to source.

This technique will not destroy chlorinated benzenes, chlorinated naphthalenes, and a number of chlorinated pesticides.

APPARATUS AND MATERIALS:

Vials: 12 mL capacity, with teflon-lined crimp cap.

Pipets: disposable 2 mL, short stem.

REAGENTS:

Hexane (Pesticide grade or equivalent).

Concentrated Sulfuric Acid (H_2SO_4)

CAUTION: SULFURIC ACID BURNS, AVOID SKIN CONTACT.

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Standard Operating Procedure
Analytical Method

TITLE: Analysis of Toxaphene

DEPARTMENT: Semivolatile Organics

APPLICATION: This method is used to determine the concentration of toxaphene in water and solid waste. Appendix A contains the detection limits in reagent water.

REFERENCES: Test Methods for Evaluating Solid Wastes
SW846 method 8000A (3rd Ed., Rev. 1, July 1992)
SW846 method 8080 (3rd Ed., Sept. 1986)

Code of Federal Regulations
USEPA method 608 40CFR Pt. 136, App. A, Ch. 1 (7-1-88 Ed.)

PROCEDURE SUMMARY:

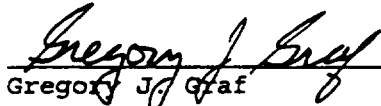
This method provides the gas chromatographic conditions for the detection of part-per-billion levels of toxaphene in water and solid waste. A volume of a sample extract is injected into a gas chromatograph (GC) and compounds in the GC effluent are detected by an electron capture detector (ECD).

REVIEWED BY:



Daniel M. Rude
Organics Group Leader

8-30-96
Date



Gregory J. Graf
Quality Assurance Officer

9-3-96
Date

APPROVED BY:



Eric L. Thomas
Laboratory Manager

9/6/96
Date

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CLEANUP PROCEDURE:

- 1 Transfer equal amounts of concentrated sulfuric acid and sample to a 10 mL vial.

CAUTION: Make sure there is no exothermic reaction or evolution of gas prior to proceeding.

- 2 Cap the vial tightly and vortex for one minute.
- 3 Allow the phases to separate. If the hexane layer is not clear, remove the majority of the extract (top layer) to a clean vial and dispose of the acid properly.
- 4 Add another aliquot of concentrated sulfuric acid, seal the cap tightly, and vortex for one minute. Allow the phases to separate.
- 5 Using a disposable pipette, transfer the extract to an injection vial.

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SAMPLE EXTRACT HANDLING AND STORAGE

Store all extracts at $4^{\circ} \pm 2^{\circ}$ C in the dark in teflon-sealed containers until analysis is complete. Sample extracts must be analyzed within 40 days from time of extraction.

INTERFERENCES:

Method interferences may be caused by contaminants (primarily phthalate esters) in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts and/or elevated baselines. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks. Contact with common plastics or rubber products must be avoided.

APPARATUS AND MATERIALS:

Gas Chromatograph:	Hewlett Packard (HP) 5890 equipped with an ECD or equivalent GC-ECD system.
GC Autosampler:	HP7673A or equivalent.
GC Integrator:	HP3396A or equivalent.
Data Processor:	TurboChrom IV or equivalent.
Printer:	HP laserjet 4M/Plus
Syringes:	10-1000 μ L Gastight syringes (Hamilton series 1000 or equivalent).
Autosampler Vials:	2 mL with crimp top caps.
Detector:	ECD (HP or equivalent).
GC Columns:	<u>Column 1</u> - DB-5 Capillary column, 30 m x 0.32 mm I.D. (J&W Scientific or equivalent). <u>Column 2</u> - DB-1701 Capillary column, 30 m x 0.32 mm I.D. (J&W Scientific or equivalent).

GC Column Conditions: Carrier gas - Helium
Flow rate - 2 mL/min.
Make-up gas - Nitrogen
Flow rate - 60 mL/min.
Detector temp. - 350° C
Injector temp. - 205° C
Splitless injection

GC Temperature Program: Initial temp. - 110° C
Initial time - 0.5 min.
Rate (1) - 20° C/min.
Hold Time (1) - 0.0 min.
Rate (2) - 11° C/min.
Final temp. - 280° C
Final time - 10 min.

REAGENTS:

Solvents: Hexane, acetone, and isooctane (2,2,4-trimethylpentane) pesticide grade.

Stock Standards Solutions: Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or an independent source. Shelf-life of standard solutions is 6 months from the date of preparation.

Surrogate Stock Standards: Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or an independent source. Shelf-life of standard solutions is 6 months from the date of preparation. See Appendix C.

Calibration Standards: Five solutions containing toxaphene and surrogate.

ANALYSIS SEQUENCE:

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The analytical sequence contains the following standards and samples.

- | | |
|--------------|---|
| 1 | A solvent blank (hexane) |
| 2 | 5 point standard curve for toxaphene |
| 3 through 12 | samples, method blanks, etc. |
| 13 | Midpoint calibration check (Midpoint toxaphene standard) etc. |

Step 2 defines the calibration curve.

Up to 10 samples, including method blanks, control spikes, and matrix spikes are analyzed between midpoint calibration check standards.

ANALYSIS ACCEPTANCE CRITERIA:

Calibration Curve criteria:

1. 5 point toxaphene curve.
 - a. Calibration factor for toxaphene.

$$CF = \frac{\text{Total average area of defined peaks}}{\text{Mass injected (in nanograms)}}$$

The percent relative standard deviation (%RSD) of the calibration factors over the working range must be less than 20% on the primary column. If the %RSD is less than 20% then linearity can be assumed and the average calibration factor can be used in place of the calibration curve. If the %RSD is greater than 20%, recalibration is required.

2. Absolute retention time.
 - a. The absolute retention times (RT) are determined for toxaphene and the surrogates during the initial calibration. See appendix C for the retention time windows.

Midpoint calibration check standard (Midpoint toxaphene standard)

1. The percent difference (%D) must be within $\pm 15\%$ of the calibration curve for the primary column.

$$\%D = \frac{R_1 - R_2}{R_1} \times 100$$

where: R_1 = Calibration factor from first analysis

R_2 = Calibration factor from succeeding analyses

SAMPLE ANALYSIS

- 1 Quantification is performed on the primary column. Confirmation is performed on the second column.

- 2 Calculations:

Aqueous samples

$$\text{Concentration } (\mu\text{g/L}) = [(A_x) (V_t) (D)] / [(CF_{1s}) (V_i)]$$

Nonaqueous samples

$$\text{Concentration } (\mu\text{g/kg}) = [(A_x) (V_t) (D)] / [(CF_{1s}) (W)]$$

where:

A_x	=	Area counts for an analyte
V_t	=	Final volume of extract in mL
D	=	Dilution factor
CF_{1s}	=	average calibration factor
V_i	=	Initial sample volume (L)
W	=	Initial sample weight (kg)

- 3 Quantification of toxaphene and surrogate is based off the average calibration factor.
- 4 Surrogate recoveries must fall within the prescribed limits (see appendix C). If a surrogate recovery fails this criteria, re-extract the sample. If an interfering peak obscures one surrogate, then that one surrogate may be excluded.

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- 5 Sample matrix spike component recoveries must fall within the prescribed limits (see appendix C) except:
- a. If the laboratory control spike analyzed in the sequence satisfies the recovery criteria specified in appendix C, no re-extraction of the sample matrix spike is required.
 - b. If a laboratory control spike was not extracted or associated with the samples, re-extraction of the sample matrix spike is required.

QUALITY CONTROL

- 1 The method blank must meet the surrogate limits (see appendix C).
If the blank fails this criteria, all of the associated samples, matrix spikes and laboratory control spikes must be re-extracted.
- 2 If the blank contains any analyte of interest above the reporting limit (see appendix A), all of the associated samples, matrix spikes, and laboratory control spikes must be re-extracted.
- 3 If the laboratory control spike does not meet all of the recovery criteria specified in appendix C, all of the associated samples and matrix spikes must be re-extracted.

Appendix A

DETECTION LIMITS
for TOXAPHENE

<u>Compound</u>	EPA ^a Method Detection Limit <u>(ug/L)</u>	En Chem ^b Method Detection Limit <u>(ug/L)</u>	En Chem ^c Reporting Limit	
			<u>Water (ug/L)</u>	<u>Soil (ug/kg)</u>
Toxaphene	0.24	0.61	5.0	170

^aU.S EPA Method 617. Organochlorine pesticides and PCBs. Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

^bMethod Detection Limit determination, USEPA 40CFR Pt.136, App.B, 1988. Method detection limits are updated periodically, the values currently in use may differ slightly from those published.

^cEn Chem Reporting Limits based on internal Method Detection Limit determinations, USEPA 40CFR Pt.136, App.B, 1988.

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Appendix B

RETENTION TIME WINDOWS for TOXAPHENE

<u>Compound</u>	<u>RT^a Window (min)</u>
Toxaphene	± 0.07
Tetrachloro-m-xylene	± 0.05
Decachlorobiphenyl	± 0.10

SAMPLE ANALYSES QUALITY CONTROL LIMITS^b

<u>Compound</u>	<u>Water ± Rec.</u>	<u>Soil ± Rec.</u>
Decachlorobiphenyl	(51-141)	(42-168)
Tetrachloro-m-xylene	(57-129)	(58-154)
Toxaphene	(60-150)*	(60-150)*

LABORATORY CONTROL SPIKE ANALYSES QUALITY CONTROL LIMITS^b

<u>Compound</u>	<u>Water ± Rec.</u>	<u>Soil ± Rec.</u>
Decachlorobiphenyl	(51-141)	(42-168)
Tetrachloro-m-xylene	(57-129)	(58-154)
Toxaphene	(60-150)*	(60-150)*

^a USEPA Contract Laboratory Program, Statement of Work for Organic Analyses, Document Number OLM01.8

^b Limits derived from 20 sample analyses, mean value ± 3SD. Control limits are updated periodically, the values currently in use may differ slightly from those shown above.

* = Advisory limits, limits yet to be determined

Standard Operating Procedure

TITLE: Extraction of Soil Samples for Organochlorine Pesticides/PCBs
DEPARTMENT: Semivolatile Organic Extractions
REFERENCES: Test Methods for Evaluating Solid Wastes
SW846 method 3550A (3rd Ed., Rev. 1, September, 1994)

PROCEDURE SUMMARY:

Approximately 30 grams of soil sample is extracted with methylene chloride/acetone using a sonifier. The methylene chloride extracts are dried and exchanged to hexane, and concentrated to a volume of 10 mL.

REVIEWED BY:



Daniel M. Rude
Group Leader
Organic Laboratory

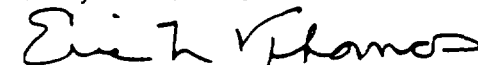
5-27-97
Date



Gregory J. Graf
Quality Assurance Officer

5-27-97
Date

APPROVED BY:



Eric L. Thomas
Laboratory Manager

5/27/97
Date

QUALITY CONTROL:

- The sample holding time is 14 days from the date of sampling
- One method blank is extracted per 20 samples OR per extraction batch whichever is more frequent. Reagent method blanks are prepared from Ottawa sand
- Surrogate standards should be added to all samples, laboratory control spikes, matrix spikes, and method blanks. Surrogates are used to monitor unusual matrix effects, sample processing problems, etc.
- A matrix spike and a matrix spike duplicate must be performed for every 20 samples. The time frame for the 20 samples cannot extend beyond 14 days. Matrix spike compounds are used to indicate the presence or absence of unusual matrix effects.
- A laboratory control spike should be performed with the matrix spike and the matrix spike duplicate for every 20 samples. The time frame for the 20 samples must not extend beyond 14 days. Control spikes are prepared from Ottawa sand.

INTERFERENCES:

Method interferences may be caused by contaminants (primarily phthalate esters) in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts and/or elevated baselines. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks. Contact with common plastics or rubber products must be avoided.

MATERIALS AND APPARATUS:

Concentrator tube:	Kuderna-Danish, 10 mL, graduated (Kontes K-570050-1025 or equivalent).
Evaporation flask:	Kuderna-Danish 300 mL (Reliance (Reliance G-9601-001 or equivalent).
Snyder column:	Kuderna-Danish, three-ball macro (Kontes K-503000-0121 or equivalent).
Vials:	Amber glass, 12 mL capacity with Teflon-lined screw cap.
Funnel:	150-gram capacity.
Boiling chips:	Solvent rinsed and dried, approximately 10/40 mesh (silicon carbide or equivalent).
Sodium Sulfate:	Preheated at 400° C for 4 hours in a crucible.
Ottawa Sand:	Preheated at 400° C for 4 hours in a crucible.

2 0 0 0 0 0 0 0 0 0

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Water bath : Heated, with concentric ring cover, capable of temperature control ($\pm 5^{\circ}\text{C}$). The bath should be used in a hood.

Tube heater: Kontes 720000-0000.

Pipets: 2 mL short-stem.

Syringes: 250-1000 μL Gastight syringes (Hamilton 1000 series or equivalent).

Branson 450 sonifier

Beaker: 250 mL.

Filter paper: Whatman #41 (or equivalent), solvent extracted

Balance: Capable of weighing 300 g \pm 0.01 g.

Spatula

REAGENTS:

Surrogate Spiking Solution: See appendix A.

Matrix Spiking Solution: See appendix B.

Methylene chloride, acetone, and hexane pesticide grade.

EXTRACTION PROCEDURE OUTLINE:

- 1 Homogenize soil sample with spatula.
- 2 Place 250 mL beaker on balance, tare balance, add 30.0 g sample to beaker. Record initial sample weight to the nearest 0.1 gram.
- 3 Add 60 g anhydrous sodium sulfate to the beaker, mix thoroughly until the mixture becomes free-flowing, i.e., dry.
- 4 Prepare method blank (and laboratory control spike if required) with Ottawa sand in place of the actual sample. Add >60 g anhydrous sodium sulfate. Mix thoroughly.
- 5 Add the required volume (200 μL) of the surrogate spiking solution to all samples, laboratory control spikes, matrix spikes, and method blanks using a 250 μL syringe. **Record the amount of spiking solution used and the reference number on the surrogate solution vial on the extraction form.**

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- 6 Add the required volume (800 μ L of Soln. 1, PCB matrix spiking solution; 400 μ L of Soln. 2, Pesticide matrix spiking solution; or, 800 μ L of Soln. 3, Toxaphene matrix spiking solution) to the appropriate QC samples using a 500 μ L or 1000 μ L syringe. **Record the amount of spiking solution used and the reference number on the matrix spiking solution vial on the extraction form.**
- 7 Add 80 mL methylene chloride/acetone (80:20) to samples. **Record the lot # of methylene chloride and acetone on the extraction form.** Sonicate sample for 3 minutes. [Set the Branson 450 Sonifier at 50 percent Duty Cycle and an Output Control setting of 5.] Filter extract through glass funnel equipped with Whatman #41 filter paper and approximately 10 g anhydrous sodium sulfate. Rinse the sodium sulfate with methylene chloride. Collect the extract in a 300 mL K-D apparatus.
- 8 Repeat step 7 two more times. If the filtration takes more than 15 minutes, apply vacuum suction to assist in the process.
- 9 In between each sample, clean the Sonifier probe with methylene chloride and clean Kimwipes to avoid any cross-contamination. Rinse the probe with methylene chloride. Do not use paper towels for cleaning the probe; interferences will result.
- 10 Attach a Snyder three-ball column to the K-D apparatus and set aside for future concentration.

SAMPLE EXTRACT CONCENTRATION:

- 1 Place the K-D apparatus on a hot water bath (80^o to 90^o C) so that the concentrator tube is totally immersed in the hot water and the entire lower rounded surface of the flask is bathed with water. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of the liquid reaches 2-5 mL, add 50 mL hexane and continue concentration to a volume of 4-6 mL. **Record the lot # for hexane on the extraction form.** Remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.
- 2 Rinse the Snyder column with 1-2 mL of hexane. Remove Snyder column and rinse the evaporating flask with 1-2 mL hexane.
- 3 Remove the evaporating flask from the concentrator tube and adjust the final extract volume to 10.0 mL with hexane.
- 4 Using a disposable pipette, transfer extract to a 12 mL amber vial.
- 5 Label the vial with project name, extraction date, sample number, and final volume.
- 6 Log sample into the analysts' extract storage refrigerator and complete all paperwork.

- 6 Add the required volume (800 μ L of Soln. 1, PCB matrix spiking solution, 400 μ L of Soln. 2, Pesticide matrix spiking solution; or, 800 μ L of Soln. 3, Toxaphene matrix spiking solution) to the appropriate QC samples using a 500 μ L or 1000 μ L syringe. **Record the amount of spiking solution used and the reference number on the matrix spiking solution vial on the extraction form.**
- 7 Add 80 mL methylene chloride/acetone (80:20) to samples. **Record the lot # of methylene chloride and acetone on the extraction form.** Sonicate sample for 3 minutes. [Set the Branson 450 Sonifier at 50 percent Duty Cycle and an Output Control setting of 5.] Filter extract through glass funnel equipped with Whatman #41 filter paper and approximately 10 g anhydrous sodium sulfate. Rinse the sodium sulfate with methylene chloride. Collect the extract in a 300 mL K-D apparatus.
- 8 Repeat step 7 two more times. If the filtration takes more than 15 minutes, apply vacuum suction to assist in the process.
- 9 In between each sample, clean the Sonifier probe with methylene chloride and clean Kimwipes to avoid any cross-contamination. Rinse the probe with methylene chloride. Do not use paper towels for cleaning the probe; interferences will result.
- 10 Attach a Snyder three-ball column to the K-D apparatus and set aside for future concentration.

SAMPLE EXTRACT CONCENTRATION

- 1 Place the K-D apparatus on a hot water bath (80^o to 90^o C) so that the concentrator tube is totally immersed in the hot water and the entire lower rounded surface of the flask is bathed with water. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of the liquid reaches 2-5 mL, add 50 mL hexane and continue concentration to a volume of 4-6 mL. **Record the lot # for hexane on the extraction form.** Remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.
- 2 Rinse the Snyder column with 1-2 mL of hexane. Remove Snyder column and rinse the evaporating flask with 1-2 mL hexane.
- 3 Remove the evaporating flask from the concentrator tube and adjust the final extract volume to 10.0 mL with hexane.
- 4 Using a disposable pipette, transfer extract to a 12 mL amber vial.
- 5 Label the vial with project name, extraction date, sample number, and final volume
- 6 Log sample into the analysts' extract storage refrigerator and complete all paperwork

Appendix B

MATRIX SPIKING SOLUTION

PCB Matrix Spike

10.0 µg/ml

Aroclor 1016, 1221, 1232, 1242, 1248, 1254, or 1260 (rotated on a semi-annual basis)

Pesticide Matrix Spike

0.5 µg/ml

g-BHC
a-BHC
Endosulfan I
Heptachlor
Aldrin
b-BHC
d-BHC
a-Chlordane
g-Chlordane
Heptachlor epoxide

1.0 µg/ml

4,4'-DDD
4,4'-DDT
Dieldrin
Endrin
4,4'-DDE
Endosulfan II
Endosulfan sulfate
Endrin aldehyde
Endrin ketone

5.0 µg/ml

Methoxychlor

Standard Operating Procedure

TITLE: Extraction of Water Samples for Organochlorine
Pesticides/PCBs

DEPARTMENT: Semivolatile Organic Extractions

REFERENCES: Test Methods for Evaluating Solid Wastes
SW846 method 3510B (3rd Ed., Rev. 2, September, 1994)
Code of Federal Regulations
USEPA 40CFR (1988), Pt.136, App.A, Method 608

PROCEDURE SUMMARY:

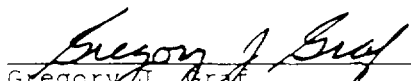
A measured volume of water sample, approximately one liter, is serially extracted with methylene chloride at a neutral pH using a separatory funnel. The methylene chloride extracts are dried and exchanged to hexane and concentrated to a volume of 10 mL.

REVIEWED BY:



Daniel M. Rude
Group Leader
Organic Laboratory

3-11-97
Date


Gregory J. Grief
Quality Assurance Officer

3-11-97
Date

APPROVED BY:


Eric L. Thomas
Laboratory Manager

3/15/97
Date

QUALITY CONTROL:

- The sample holding time is 7 days from date of sampling.
- One method blank is extracted per 20 samples OR per extraction batch whichever is more frequent. Reagent method blanks are prepared from laboratory de-ionized water.
- Surrogate standards should be added to all samples, laboratory control spikes, matrix spikes, and method blanks. Surrogates are used to monitor unusual matrix effects, sample processing problems, etc.
- A matrix spike and a matrix spike duplicate will be performed for every 20 samples. The time frame for the 20 samples cannot extend beyond 14 days. If unsufficient volume or matrix problems do not allow this, two laboratory control spikes may be prepared instead. Matrix spike compounds are used to indicate the presence or absence of unusual matrix effects.
- A laboratory control spike should be performed with the matrix spike and the matrix spike duplicate for every 20 samples. The time frame for the 20 samples cannot extend beyond 14 days. Control spikes are prepared from laboratory de-ionized water.

INTERFERENCES:

Method interferences may be caused by contaminants (primarily phthalate esters) in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts and/or elevated baselines. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks. Contact with common plastics or rubber products must be avoided.

MATERIALS AND APPARATUS:

Separatory funnel:	2000 mL with Teflon stopcock.
Concentrator tube:	Kuderna-Danish, 10 mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be demonstrated prior to use.
Evaporation flask:	Kuderna-Danish 300 mL (Reliance G-9601-001 or equivalent). Attach to concentrator tube with clips.
Snyder column:	Kuderna-Danish, three-ball macro (Kontes K-503000-0121 or equivalent).
Vials:	Amber glass, 12 mL capacity with teflon-lined screw cap.
Funnel:	150-gram capacity.

Glass wool: Rinsed with methylene chloride.

Boiling chips: Solvent rinsed and dried, approximately 10/40 mesh (silicon carbide or equivalent).

Sodium Sulfate: Preheated at 400° C for 4 hours in a crucible.

Water bath: Heated, with concentric ring cover, capable of temperature control ($\pm 5^{\circ}$ C). The bath should be used in a hood.

Tube heater: Kontes 720000-0000.

Pipets: Disposable, 2 mL short-stem.

Syringes: 250-1000 L Gastight syringes (Hamilton 1000 series or equivalent).

Graduated Cylinder: 1000 mL.

pH paper: Wide range pH paper.

REAGENTS:

Surrogate Spiking Solution: See appendix A.

Matrix Spiking Solution: See appendix B.

Sodium hydroxide (NaOH): 10 Normal; dissolve 80 g of NaOH pellets in reagent water and dilute to 200 mL.

Sulfuric Acid (H₂SO₄): (1:1); slowly add 50 mL concentrated H₂SO₄ (specific gravity 1.84) to 50 mL reagent water.

Methylene chloride, acetone, and hexane pesticide grade.

EXTRACTION PROCEDURE OUTLINE:

- 1 Allow the sample to warm to room temperature, mark the sample volume on the sample container, then invert sample several times.
- 2 Set up and label separatory funnel, K-D apparatus, and funnel with glass wool and sodium sulfate.
- 3 Transfer sample volume to separatory funnel.
- 4 Add 60 mL methylene chloride to sample container, swirl container several times, then transfer solvent to separatory funnel. **Record methylene chloride lot # on the extraction form.**
- 5 Add the required amount (200 μ L) of surrogate solution to each separatory funnel with a 250 μ L syringe. **Record the amount of surrogate solution used and the reference number on the surrogate solution vial on the extraction form.**
- 6 Add the required volume (400 μ L of the pesticide matrix spiking solution or 1000 μ L of the PCB matrix spiking solution) to only those required samples using a 500 or 1000 μ L syringe. **Record the amount of spiking solution used and the reference number on the matrix spiking solution vial on the extraction form.**
- 7 Adjust sample pH between 5-9 with either 5 N sodium hydroxide or sulfuric acid (1:1). Check with wide range pH paper.
- 8 Shake separatory funnel for 1 to 2 minutes, with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase. If an emulsion interface occurs between the phases, the technician must employ mechanical techniques to complete phase separation.
NOTE: Methylene chloride creates excessive pressure very rapidly: therefore, initial venting should be done immediately after the separatory funnel has been sealed and inverted. Venting of the separatory funnel should be into a hood to avoid needless exposure of the technician to solvent vapors.
- 9 Drain the extract through the funnel containing glass wool and sodium sulfate. Collect the extract in the K-D apparatus. Rinse the sodium sulfate with methylene chloride.
- 10 Add 60 mL methylene chloride to the separatory funnel and repeat steps 8 through 9. This process is repeated one more time. The total extract volume in the K-D apparatus should be approximately 200 mL.
- 11 Attach a Snyder three-ball column to the K-D apparatus and set aside for future concentration of sample extract.
- 12 Determine initial sample volume. Fill the original sample container from step 1 to the mark with water. Transfer contents to a 1000 mL graduated cylinder. Record the initial sample volume on the extraction form to the nearest 5 mL.

SAMPLE EXTRACT CONCENTRATION:

- 1 Place the K-D apparatus on a hot water bath (80 to 90 C) so that the concentrator tube is totally immersed in the hot water and the entire lower rounded surface of the flask is bathed with water. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of the liquid reaches 2-5 mL, add 50 mL hexane and continue concentration to an apparent volume of 4-6 mL. **Record the lot # for hexane on the extraction form.** Remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.
- 2 Rinse the Snyder column with 1-2 mL of hexane. Remove Snyder column and rinse the evaporating flask with 1-2 mL hexane.
- 3 Remove the evaporating flask from the concentrator tube and adjust the final extract volume to 10.0 mL with hexane.
- 4 Using a disposable pipette, transfer extract to a 12 mL amber vial.
- 5 Label the vial with project name, extraction date, sample number, and final volume.
- 6 Log sample into the analysts' extract storage refrigerator and complete all paperwork.

Appendix A

SPIKING STANDARDS FOR PESTICIDES/PCBS**SURROGATE SPIKING SOLUTION**

The mixture contains the following components at:

2.0 µg/mL
DCB (decachlorobiphenyl)
TCMX (tetrachloro-m-xylene)

Appendix B

MATRIX SPIKING SOLUTION

PCB Matrix Spike

10.0 µg/mL
Aroclor 1016, 1221, 1232, 1242, 1248, 1254, or 1260 (rotated
on a semi-annual basis)

Pesticide Matrix Spike

0.5 µg/mL
γ-BHC
α-BHC
Endosulfan I
Heptachlor
Aldrin
β-BHC
δ-BHC
α-Chlordane
γ-Chlordane
Heptachlor epoxide

1.0 µg/mL
4,4'-DDD
4,4'-DDT
Dieldrin
Endrin
4,4'-DDE
Endosulfan II
Endosulfan sulfate
Endrin aldehyde
Endrin ketone

5.0 µg/mL
Methoxychlor

August 14, 1997

PROCEDURE FOR THE DETERMINATION OF TOXAPHENE

This analysis plan describes the method to be used by the U.S. Environmental Protection Agency, Region IV Laboratory, Athens, Georgia, (EPA) and Hercules Incorporated, through its contract laboratory, En Chem, Inc., Madison, Wisconsin, (En Chem) for the determination of toxaphene in soil, sediment, and water samples from the Brunswick, Georgia, area.

All analyses will be performed according to approved EPA SW-846 Method 8081 -- for the liquid-liquid extraction of water samples, for the ultrasonic extraction of the soil and sediment samples, and for the gas chromatography (GC) with electron capture detector (ECD) analyses of the extracts. Because the EPA and Hercules will be analyzing split samples, certain aspects of the methods have been specified in detail to assure consistent application of the methods. All QA/QC requirements specified in the methods will be followed by the laboratories.

I.) TOXAPHENE REFERENCE STANDARD:

All laboratories must use a toxaphene reference standard which matches the GC profile of the Hercules product standard, that is, Hercules technical toxaphene X16189-49. Presently, three commercial sources -- AccuStandard, Chem Service, and Restek -- can supply satisfactory toxaphene standards. However, the match with the Hercules technical toxaphene must be established each time a new lot of standard is purchased.

II.) ANALYTICAL METHOD:

U.S. EPA SW-846 Method 8081, "Organochlorine Pesticides, Halowaxes, and PCBs as Aroclors by Gas Chromatography: Capillary Column Technique," with a sulfuric acid clean up, will be followed for all analyses. For consistent application of the method between laboratories, the following steps are specified in more detail.

1.) GC Columns:

- a.) 30-meter DB-1701 (1.0- μ m film thickness) Megabore (J&W Scientific).
- b.) 30-meter DB-5 (1.5- μ m film thickness) Megabore (J&W Scientific).

NOTE: Use the DB-1701 column for quantitation where possible, that is, when there are fewer interfering peaks on the DB-1701 column than on the DB-5 column.

August 14, 1997

2.) GC Column Oven Temperature Program:

The column oven temperature profile will be adjusted in each laboratory to separate the GC peaks satisfactorily and to demonstrate the performance criteria required in Method 8081.

3.) GC Operating Temperatures:

Injector: 220°C - 250°C

Electron Capture Detector: 300°C - 350°C

- 4.) Tetrachloro-m-xylene (TCMX) and/or decachlorobiphenyl (DCB) will be used as surrogates.
- 5.) A five-point toxaphene calibration curve will be used.
- 6.) A continuing calibration sample will be injected after every 10 samples; and, as the final injection at the end of each injection sequence.
- 7.) Four to seven major peaks in the "back half" of the toxaphene chromatogram will be used for calibration and quantitation of toxaphene. The "back half" of the chromatogram is defined as the major peak in the toxaphene chromatogram and all peaks with longer retention times.
- 8.) The Perkin-Elmer TurboChrom data system will be used for measurement of peak heights. Baseline parameters will be selected so that baselines are drawn by the TurboChrom data system from valley-to-valley under the groups of peaks in the chromatogram so that the resulting baselines follow the general curve of the toxaphene baseline. (See Figure 1.) Adjust the baselines with manually-selected baselines (denoted by M in the TurboChrom printout of the chromatogram) if the data system selects a peak shoulder or other erroneous baseline.
- 9.) The baselines in the chromatograms of the samples will be placed under the peaks exactly as in the calibration standards (Steps 7 and 8, above).
- 10.) Peaks which are larger in relative proportions in the samples than in the toxaphene standard will not be used for quantitation.
- 11.) If fewer than four peaks are used for quantitation, the resulting values will be considered only as estimated values; and those results will be qualified as "J" values.

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- 12.) Any results qualified as "JN" will be interpreted to mean "not detected," or "U" values.

III.) EXTRACTION OF SAMPLES:

U.S. EPA SW-846 Method 3550A, "Ultrasonic Extraction," (SW-846) will be used for the extraction of all soil and sediment samples. The extraction solvent will be a 1:1 mixture of hexane and acetone. All solvent extracts will be exchanged to hexane for analysis by GC. The extracts will be made to a final volume of 10 mL.

U.S. EPA SW-846 methods will be used for the liquid-liquid extraction of all water samples. The extraction solvent will be methylene chloride. All solvent extracts will be exchanged to hexane for analysis by GC. The extracts will be made to a final volume of 10 mL.

NOTE: The final volume of the sample extracts may be adjusted according to the detection limit required by the data quality objectives specified in the specific project plan.

IV.) SULFURIC ACID CLEANUP:

After the sample extract is adjusted to final volume in hexane, add 10 mL of concentrated sulfuric acid for each 5 mL of sample extract in a glass vial with a Teflon-lined screw cap. Shake the tube vigorously for one minute. Vent the vial carefully to relieve any pressure that may build up in the vial. Allow the layers to separate. (If excessive heat is generated during the extraction, the sample extract should be discarded and steps taken to eliminate the source of the heat generation.) If the layers are not clearly separated, centrifuge the mixture. After a clear separation is obtained, transfer the hexane layer to a GC injection vial for analysis. Store the excess extract in a clean vial with a Teflon-lined screw cap in a refrigerator at 4°C, or less.

V.) LABORATORY COMPLIANCE:

Lavon Revells will be responsible for assuring that the U.S. EPA Region IV Laboratory complies with this methodology, and F. J. Carlin will be responsible for compliance at Hercules Incorporated and its contract laboratory.

TABLE 1

**CHEMICAL AND PHYSICAL PROPERTIES OF
CONSTITUENTS OF CONCERN**

Contaminant	Flash Point (°C)	Upper Explosive Limit	Lower Explosive Limit	IDLH (mg/m³)	Ionization Potential (eV)	Density (g/cm³)
Toxaphene	34.4 and 135 (closed cup)	NA	NA	200	NA	1.65 at 25°C

APPENDIX D

GEOSYNTEC CONSULTANTS

GEOMECHANICS AND ENVIRONMENTAL

LABORATORY

STANDARD OPERATING PROCEDURES

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General Lab Procedure Title: Sample Log-in and Handling Procedure

GEL Author(s) of Procedure: James Stalcup

Approval By:

Reference No.: N/A

Material Applicability: All test material samples

Goal of Procedure: Accurate sample tracking and documentation

Remarks:

Standard Operating Procedures:

1. Non-contaminated Samples (G-LAB)
 - A. Upon receipt of the bulk sample to be tested in the laboratory, determine what project owns the sample. A Laboratory Test Request form should accompany all Geosyntec project samples.
 - B. Inspect the sample packaging for damage during shipment.
 - D. If the sample is for environmental testing, go to section 2. Contaminated Samples (E-LAB).
 - D. Open the container, provided that it is known that the sample is NOT contaminated, and inspect the contents then re-seal the container.
 - E. Determine the client sample ID.
 - F. Determine the required testing.
 - G. Open the G-LAB Sample Log-in Book to the last entry and record the necessary information and assign a laboratory sample number to the sample (see the attached example).
 - H. Attach or write the sample number and project number on the sample container. One of the red and white stickers with the sample and project number should be used to mark the container.
 - I. Fill out a summary sheet and the necessary data sheets, then place them in the holding boxes for testing.

- J. Place the container into the temporary storage area if testing is to begin upon receipt or into the general storage area until the sample is needed for test specimen sampling.
2. Contaminated Samples (E-LAB)
- A. Upon receipt of the bulk sample to be tested in the laboratory, determine what project owns the sample. A Laboratory Test Request form should accompany all GeoSyntec project samples.
 - B. Inspect the sample packaging for damage during shipment.
 - C. Open the container, provided that proper safety precautions are followed (i.e.; use the fume hood, gloves, etc.), and inspect the contents, then re-seal the container.
 - D. Determine the client sample ID.
 - E. Determine the required testing.
 - F. Open the E-LAB Sample Log-in Book to the last entry and record the necessary information and assign a laboratory sample number to the sample (see the attached example).
 - G. Attach or write the sample number and project number on the sample container. One of the fluorescent orange stickers with the sample and project number should be used to mark the container.
 - H. Fill out a summary sheet and the necessary data sheets, and place them in the holding boxes for testing.
 - I. Sample storage must be done so that incompatible materials are separate (see Cuneyt Gokmen for questions).
 - J. Place the container into the temporary storage area if testing is to begin upon receipt or into the general storage area until the sample is needed for test specimen sampling.
3. Composite Samples and Altered Samples
- A. Composite Samples: Occasionally composite mixtures of two or more samples are made at the request of the client. When this occurs, use the following procedures:
 - 1. If possible, retain approximately 500g or more of the individual samples to be mixed, prior to mixing.
 - 2. Mix the samples following the client's specifications.
 - 3. Assign a new Lab Sample No. using procedures similar to those presented above.
 - 4. Mark the composite sample container with the new number.

- B. Altered Samples: Occasionally samples are altered at the request of the client. Alterations are usually made to the grain-size distribution of the sample. When alterations are made to the sample which are not specified in a testing standard, use the following procedure:
1. If possible, retain approximately 500g or more of the individual samples to be mixed, prior to alteration.
 2. Conduct the client specified alteration.
 3. Assign a new Lab Sample No. using procedures similar to those presented above.
 4. Mark the altered sample container with the new number.

General Lab Procedure Title: Sample Storage Procedure

GEL Author(s) of Procedure: James Stalcup

Approval By:

Reference No.:

Material Applicability: All test material samples

Goal of Procedure: Sample Location

Remarks: Alternate procedures may be required for very large or special samples.

Standard Operating Procedures:

1. Determine what section of the laboratory owns the sample (i.e., G-Lab or E-Lab) and determine if the sample is contaminated.
2. Place the sample into the proper storage area using the following guidelines -- making sure that the sample container is well sealed and well labeled and that material incompatibility does NOT exist (see Environmental Health and Safety Manager (EHSM) for compatability questions):
 - A. Contaminated samples are stored in the E-Lab Storage area while testing and sampling are being performed and may remain in this area until returned to the client.
 - B. Contaminated samples may be stored in the E-Lab Storage Room after testing and sampling have been completed if approved by the EHSM.
 - C. Uncontaminated samples are stored in the G-Lab temporary storage area while testing and sampling are being performed.
 1. Buckets will be stacked on and under the large shelves in the proctor/sample preparation area up to 3 buckets high.
 2. Shelby tubes will be placed on the shelves near the roll-up door in the proctor/sample preparation area.
 3. Other samples will be stored on the shelves or in the floor depending on size.
 - D. Uncontaminated samples are moved to the G-Lab Storage Room when the testing and sampling have been completed.
 1. Buckets will be stacked along the left wall of the storage room chronologically from back to front up to 4 buckets high.

2. Shelby tubes will be placed on the shelves along the right side of the storage room.
3. Other samples will be stored on the shelves or in the floor depending on size.

General Lab Procedure Title: General Procedures for Sampling

GEL Author(s) of Procedure: James Stalcup

Approval By:

Reference No.:

Material Applicability: All material to be sampled for testing

Goal of Procedure: Representative Test Samples

Remarks: Sample size may pose some problems in proper sampling. Consult project manager when needed.

Standard Operating Procedures:

1. Review the required testing and check total sample size to make sure enough material has been provided for the required tests.
2. Using the attached Required Amount of Material for Testing Table, obtain the required amount of material needed for the test by quartering or using the sample splitter.

A. Method of Quartering

1. Place sample on a clean flat surface.
2. Using a shovel or hand scoop, mix the sample thoroughly by turning the entire sample over three times.
3. Scoop material into a conical pile.
4. Flatten the pile.
5. Divide the pile into four pie-shaped sections through the center.
6. Combine opposing sections to yield two representative samples.
7. Continue the above procedures on one of the two samples until the desired amount of material is obtained.

B. Sample Splitter

1. Moist soil does not usually work well using the splitter.

2. Set the splitter openings approximately 50% larger than the largest particle size in the sample.
3. Make sure the catch pans are in place and close the chute.
4. Pour sample into the chute.
5. Slowly, open the chute, letting the sample pass through to the catch pans.
6. Repeat the above procedures using the splitter until the desired amount of material is obtained.
7. For repeat splitting, the operator should alternate catch pan usage for best results required amount of material for testing.

Testing	Required Amount		Remarks
	Material Type	Specimen Size (grams)	
Moisture Content ASTM D 2216 ASTM D4643	Fine		
	Sandy		
	Coarse		
Sieve ASTM D 422(1)	Fine	200	oven dried
	Sandy	500	oven dried
	Coarse	2000	oven dried
Sieve ASTM C 136(2)	Fine	300	oven dried
	Sandy	1000	oven dried
	Coarse	5000+	oven dried
Hydrometer ASTM D 422	Fine	100	air-dried
	Sandy	150	air-dried
	Coarse		usually not required
Atterberg Limits ASTM D 4318	Fine	200	soaked in DI water or air-dried
	Sandy	250	soaked in DI water or air-dried
	Coarse	300+	soaked in DI water or air-dried
Proctor Compaction	Fine		

ASTM D 698	Sandy		
ASTM D 1557	Coarse		
Relative Density	Fine		
ASTM D4253/4254	Sandy		
	Coarse		
Specific Gravity	Fine	100	usually testing is done on moist soil
ASTM D 854	Sandy	200	usually testing is done on moist soil
	Coarse		see alternate testing method
Specific Gravity	Sandy	2000	
ASTM C 127(3)	Coarse	3000+	
Carbonate Content	Fine	600	oven dried, usually triplicate specimens
ASTM D 3042	Sandy	1500	oven dried, usually triplicate specimens
	Coarse	1500	oven dried, usually triplicate specimens
Calcium Carbonate Content	Fine	20 - 30	oven dried
ASTM D 4373	Sandy	20 - 30	oven dried
	Coarse	20 - 30	oven dried
Free Swell	Fine	15	oven dried
ASTM D 5890	Sandy	50 - 100	oven dried
	Coarse	100+	oven dried
Permeability FWP	Fine	800	place in bag and obtain moisture content
ASTM D 5084	Sandy	800	place in bag and obtain moisture content
	Coarse	test dependent	
Permeability RWP	Fine	4000	place in bag and obtain moisture content
ASTM D2434	Sandy	4000	place in bag and obtain moisture content
	Coarse	see project manager	
Gradient Ratio	Fine	2000	Place in bag and obtain moisture content
ASTM D5101	Sandy	2000	Place in bag and obtain moisture content
	Coarse	test dependent	
	Fine	800	Place in bag and obtain moisture content

HCR ASTM D5567	Sandy	800	Place in bag and obtain moisture content
	Coarse	testdependent	
Triaxial Shear ASTM D4767	Fine	4500	Usually 3 specimens, 1500g each Place in bag and obtain moisture content
	Sandy	4500	Usually 3 specimens, 1500g each Place in bag and obtain moisture content
	Coarse	test dependent	Usually 3 specimens, 1500g each
Ring Shear	Fine	500	
	Sandy	500	
	Coarse	N/A	
One-Dimensional Consolidation ASTM D2435	Fine	800	
	Sandy	800	
	Coarse	N/A	
Mass/Area ASTM NEW			
Soil Organic content or LOA	Fine		Usually conducted on moisture content specimen.
	Sandy		Usually conducted on moisture content specimen.
	Coarse		Usually conducted on moisture content specimen.

Note:

1. Refer to section 5.1.1 of the test standard for more specific sample size.
2. Refer to section 7.4 of the test standard for more specific sample size.
3. Refer to section 7.3 of the test standard for more specific sample size.

General Lab Procedure Title: Quality Assurance/Quality Control (QA/QC) Protocol

GEL Author(s) of Procedure: Barry Sigmon/James Stalcup

Approval By:

Reference No.:

Material Applicability: All testing data sheets

Goal of Procedure: Correct and Accurate Documentation

Remarks: More specific QA/QC procedures are provided in the Quality Assurance/Quality Control (QA/QC) for Test Data and the QA/QC for laboratory equipment's SOP.

Standard Operating Procedures:

1. Technicians - Testing Information
 - A. Use a pencil to perform all work. ALL DATA ENTRY SECTIONS MUST BE FILLED IN. If the information does not apply put a line through the blank. Initial and date each page.
2. Checkers - Initial QA/QC
 - A. Use a red pen to check all calculations.
 - B. Initial each page and date.
 - C. Place a red check next to each major correct calculation/result checked.
 - D. Draw a single red line through each incorrect calculation or result and write the correct information in red.
 - E. If no corrections were made then go to the project box on the wall; place test data onto the appropriate summary table; place a diagonal line (/) thru the test date on the larger table; when the data has been reported, a 2nd line (X) is put thru the date.
 - F. If a calculation is incorrect, return paperwork to technician to review their error.
3. Technicians - Data/Results in error
 - A. Using a blue pen, review the work of checker in the event that errors (items in red) were corrected during the initial QA/QC.

- B. With a blue pen place a check next to information which has been corrected.
 - C. Draw a single line through any entry which is incorrect and write the correct information in blue.
 - D. Initial and date pages.
 - E. If the data calculations are correct follow the procedure of section B, number 5.
 - F. If further errors were found, return paperwork to the checker to review the error.
4. Checkers - Returned QA/QC errors
- A. Use a green pen to review all work.
 - B. Review technicians work if changes have been made to information in red (marked in blue).
 - C. Place a green check next to correct information, initial and date.
 - D. If information is not correct get together with technician to try to resolve the correction. If not able to resolve then consult with the Laboratory Program Manager.

General Lab Procedure Title: QA/QC for Test Data

GEL Author(s) of Procedure: Scott Shipley/James Stalcup

Approval By:

Reference No.:

Material Applicability: All testing data sheets

Goal of Procedure: Correct and Accurate Test Data

Remarks:

Standard Operating Procedures:

1. General Procedure
 - A. See attached data sheets for reference.
 - B. Check the following for accuracy against the Laboratory Test Summary Sheet:
 - Project Name
 - Project Number
 - Site ID
 - Sample Number
 - C. If above information does not match the Summary Sheet, assume the Summary Sheet is correct and change data sheet to match. BE SURE DATA SHEET IS WITH CORRECT SUMMARY SHEET BEFORE CHANGING INFORMATION.
 - D. After checking data sheets [see Quality Assurance/Quality Control (QA/QC) Protocol SOP], enter correct test results onto Summary Sheet and the project summary table.
 - E. If any corrections are needed follow the procedures provided in the Quality Assurance/Quality Control (QA/QC) Protocol SOP.
 - F. Return the paper work to the appropriate location for recalculation, if necessary (i.e., back to the gINT box).
2. Proctor Compaction (see attached sample data sheets for reference)
 - A. Check all highlighted data points on gINT data sheet against raw data sheet.
 - B. Check all highlighted data points on gINT figure against raw data sheet.

- C. Verify that all notes are accurate, check with technician if needed.
 - D. Draw in proctor curve and verify Maximum Dry Unit Weight and Optimum Moisture Content. If not correct, draw one line through incorrect numbers and write correct information.
3. Sieve Analysis (see attached sample data sheets for reference)
- A. Check all highlighted data points on gINT data sheet against raw data sheet.
 - B. Check all highlighted data points on gINT sieve figure against gINT data sheet.
 - C. Check sieve curve to insure curve is logical.
 - D. Check project information for accuracy.
 - E. Verify that all notes are accurate, check with technician if needed.
4. Hydrometer Testing (see attached sample data sheets for reference)
- A. Check all highlighted data points on gINT data sheet against raw data sheet.
 - B. Check all highlighted data points on gINT sieve figure against gINT data sheet.
 - C. Check sieve curve to insure curve is.
 - D. Check project information for accuracy.
 - E. Verify that all notes are accurate, check with technician if needed.
5. Atterberg Limits Determination (see attached sample data sheets for reference)
- A. Check all highlighted data points on gINT data sheet against raw data sheet. Insure water content percentages are within $\pm 2\%$ for the plastic limit.
 - B. Subtract plastic limit from liquid limit to get plasticity index. Check gINT sieve curve to insure values are correct.
6. Triaxial Shear Testing (see attached sample data sheets for reference)
- A. Check all highlighted data points on the computer printouts against the raw data.
 - B. Using the equations provide on the attached sample data sheet, check/calculate one row of the spreadsheet.
 - C. Plot multiple tests together and evaluate ϕ and c .

7. Mass Per Unit Area Testing (see attached sample data sheets for reference)
- A. Check all highlighted data points on the computer calculated data sheet against raw data sheet.
 - B. Verify (with manufacturer or project manager) that the geotextile mass per unit area is correct for the product being tested.
 - C. If any one moisture content is $\pm 4\%$ from the calculated average, then this moisture content should be voided; the remaining specimens should then be recalculated for a new average. Average mass per unit area will also need to be recalculated.
 - D. Recalculated values need to be checked, either by computer or by another checker.

Test Method Title: Sieve Analysis

GEL Author(s) of Procedure: Kinney Wilson/James Stalcup

Approval By:

Reference No.: ASTM D 422

Test Category: Physical test method

Material Applicability: Soil, aggregate, and soil-like materials

Target Property: Particle size

Units of Test Results: Percent finer by weight

Test Specimen Size: Dependent upon maximum particle size

No. of Specimens Tested: One

Test Equipment: Set of sieves, balance, wire brush, drying oven, soil washing equipment, and sieve shaker

Test Equipment Calibration: See the Laboratory Equipment Calibration and Maintenance Record binder

Deviations: Any deviations from the standard testing procedure shall be documented on the data sheet(s) and, if requested, reported in the final report submitted to the client.

Standard Operating Procedures:

1. Obtain a representative specimen of the soil sample. The quantity of specimen shall be determined based on the nominal diameter of the largest particles, see section 5.1.1 of ASTM D 422.
2. Dry the sample in an oven at a temperature of 110 degrees C. to + or - 5 degrees C. until the specimen reaches a constant mass (usually 16-hours).
3. Weigh and record the dry weight of the sample before the No. 200 wash.
4. Wash the sample over the No. 200 sieve until the water leaving the sieve is clear.
5. Dry the sample in an oven at a temperature of 110 degrees C. + or - 5 degrees C. until the sample reaches a constant mass (usually 16-hours).

6. Pour the sample into the set of sieves making sure that they are in the proper order (i.e., largest sieve opening size on top becoming progressively smaller).
7. Place the sieve set into a shaker making sure that the lid is in place and that the sieve set is firmly held in the shaker.
8. Run the shaker for 8 to 10 minutes and remove the set of sieves when the shaking has ended.
9. Weigh and record the cumulative weight of the test material retained for each sieve.
10. Compare the total cumulative weight with the after wash weight and make sure that the difference is within the range specified in the notes section of the test form and determine if the amount of test material passing the #200 sieve is within the specified range.
11. Place the sieved material into a plastic bag and label it with the Lab Sample No.
12. Reduce the data and generate the final graphs using gINT.

Test Method Title: Hydrometer Analysis

GEL Author of Procedure: Brent A. McDaniel/James Stalcup

Approval By:

Reference No.: ASTM D-422

Test Category: Physical test method

Material Applicability: Soils and soil-like material passing No. 200 (75-micron) sieve

Target Property: Distribution of particles smaller than No. 200 (75-micron) in soils

Units of Test Results: Percentage finer by weight

Test Specimen Size: Minimum 30g passing No. 200 (75-micron) sieve

No. of Specimens Tested: 1

Test Equipment: Scale, No. 200 (75 micron) sieve, mortar with rubber covered pestle, 151 H for measuring specific gravity in suspension, sedimentation cylinder marked for a volume of 1000 ml, stirring device (blender with a metal blade), thermometer, graduated cylinder, dry box, timing device, dispersing agent (sodium hexametaphosphate used in distilled water at a ratio of 40 g sodium hexametaphosphate per 1000 ml of solution)

Test Equipment Calibration: See the laboratory Equipment Calibration and Maintenance Record binder

Test Speed: N/A

Deviations: Any deviations from the standard testing procedure shall be documented on the data sheet(s) and, if requested, reported in the final report submitted to the client.

Standard Operating Procedures:

1. Obtain a representative soil sample by method of quartering or by using the sample splitter. The size of the sample shall be approximately 100 g for silt and clay soils and 150 g for sandy soils.
2. Place sample in dry box overnight.
3. Remove air-dried sample from box and grind using mortar and rubber-covered pestle.

4. Sieve ground material using No. 200 (75-micron) sieve being careful to collect all material passing sieve in pan. The amount passing should be no less than 30 g. If 30 g is not obtained at the end of sieve process then a larger sample must be used starting again with step number 1.
5. From the material passing No. 200 (75-micron) sieve:
 - A. Weigh and record a portion of the material minimum 20 g and place in gray mixing cup. Add 125 ml of dispersing agent solution into the gray cup. Making sure all material is submerged, allow cup containing soil and dispersing agent to soak for at least 16 hours. This material will be used in the hydrometer suspension.
 - B. From the remainder of the sample, weigh out 10 to 15 g in small metal tare and record the mass. Place in the oven overnight. This is used for hydrometer moisture correction.
6. After the soil and dispersing agent have soaked for at least 16 hours, disperse the sample further by blending slurry (soil + dispersing agent in gray cup) with the blender for at least 1 minute. If there is not enough liquid in cup for proper dispersion, deionized (distilled) water may be added to the cup and the slurry. Immediately after dispersion, remove blender from cup, being careful to wash all slurry residue off of the blender and back into the cup.
7. Pour slurry into glass sedimentation cylinder, making sure all of the residue from the gray cup is washed into the cylinder. Now add demineralized water to the cylinder until the total volume is approximately 1000 ml.
8. Using a stirring rod (hydrometer plunger), disperse sediment, starting at the bottom of the cylinder, throughout the cylinder for a period of at least 1 minute to complete the agitation of the slurry. At the end of agitation, place the cylinder in a secure, convenient location and begin timing. Take hydrometer readings at 2, 5, 15, 30, 60, 120, 250, and 1440 minutes.

Note: When taking hydrometer readings, carefully and slowly insert the hydrometer into the suspension about 25 seconds before the reading to the approximate depth it will have when the reading is taken. Readings shall be taken at the top of the meniscus formed by the suspension around the stem. Immediately after the reading is taken, carefully and slowly remove the hydrometer and place it with a spinning motion in a container of clean demineralized water.
9. After the 15 minute reading has been taken, use a thermometer to obtain the specimen solution temperature; the temperature shall also be obtained after each subsequent reading.
10. A reference sample is prepared by adding 125 ml of dispersion solution to a clean glass sedimentation cylinder and then adding demineralized water until the total volume is approximately 1000 ml. Measure and record the zero correction hydrometer reading from the reference solution.
11. Remove the small hydroscopic sample from the oven and weigh it. Subtract the tare weight from this weight (this gives you the dry weight of soil). Also subtract the tare weight from the original weight when it was placed in the oven (step 6, this now gives you the wet weight of soil). Divide the dry weight of soil by the wet weight of soil (remember, no tare); this value is the hydroscopic

moisture correction factor. Multiply this factor by the mass of the sample placed in the sedimentation cylinder to get the corrected dry mass of soil tested.

12. Input the test data into gINT for data analysis.

Test Method Title: Atterberg Limits Determination

GEL Author(s) of Procedure: Tiffany Dickey/James Stalcup

Approval By:

Reference No.: ASTM D 4318

Test Category: Physical test method

Material Applicability: Fine-grained soil and soil-like materials passing a N. 40 (0.425 mm) sieve

Target Property: Liquid limit, plastic limit and plasticity index

Units of Test Results: Unitless whole numbers (calculated as moisture contents)

Test Specimen Size: Approximately 250 g

No. of Specimens Tested: In general, 3 for liquid limit and 2 for plastic limit

Test Equipment: Glass plate, metal spatula, rubber spatula, grooving tool, 1.0 g tares, hand-operated liquid limit device, 1-bar porous plate, pressure chamber, fan

Test Equipment Calibration: See the Laboratory Calibration and Maintenance Record binder

Test Speed: N/A

Deviations: Any deviations from the standard testing procedure shall be documented on the data sheet(s) and, if requested, reported in the final report submitted to the client.

Standard Operating Procedures:

1. Obtain a representative sample of the test material (approximately 250 g).
2. Place the specimen into a gray mixing cup with deionized water.
3. Blend the slurry with the mixer until all of the sample is thoroughly mixed and broken up.
4. Run the slurry through a No. 40 (0.425 mm) sieve, use spray bottle (deionized water) to help pass slurry through No. 40 sieve.

5. Pour material passing the No. 40 sieve onto a porous plate. Connect the porous plate to vacuum. Stir the material slurry occasionally to speed the drying.
6. Remove sample from the porous plate when soil is near its liquid limit. Place the material into an Atterberg Limit container, mix the material thoroughly, and seal.
7. Using metal spatula, mix sample very well.
8. Plastic Limit:
 - A. Take about 20 g of the material and place it to dry on the plastic limit glass.
 - B. Take about half of this sample and roll into a ball between your fingers.
 - C. Roll the ball out on the glass plate into a uniform thread about 1/8 of an inch in diameter (compare with 1/8 inch reference rod). Then roll back into a ball between hands.
 - D. Continue steps B and C, alternating between halves of the sample, until the part of the thread being rolled begins to fracture (see Figure). When the specimen cannot be rolled to 1/8", the specimen has reached its plastic limit. Note: Some soils will tend to break, due to using too much finger pressure, at a diameter larger than 1/8" and the thread becomes uniform (un-broken) when rolled smaller. Do not take samples for plastic limit moisture until the thread is breaking near 1/8" diameter.
 - E. Immediately place the specimen rolled to its plastic limit into a 1.0 g tare, weigh and record the weight, and place in the oven.
 - F. Repeat steps to generate at least two approximately 3-5 g (or more) plastic limit moisture samples.
9. Liquid limit:
 - A. Check the liquid limit device: check drop distance of the cup using the calibration block, make sure nuts are tight, make sure cup is secure, etc.
 - B. Add water (de-ionized) to the material to get the moisture near liquid limit, mixing thoroughly each time water is added.
 - C. Spoon some of the sample into the liquid limit device.
 - D. Strike a groove in the soil pat.
 - E. Be sure the counter is set to zero, turn the crank (or turn on the device) using a rate of two blows per second to determine how many blows it takes to close the groove 1/2 inch.
 - F. With the above steps, try to find the moisture where approximately 25-35 blows will close the groove. Add water or dry the material as necessary.

- G. Take a moisture content once you reach 25 to 35 blows, scoop out approximately 10-30 g of the sample (from the middle in order to include the part of the sample that closed up) with the rubber spatula.
 - H. Add water (de-ionized) to the sample, mix thoroughly, and, using the above procedures, take moistures where 20-30 blows and 10-20 blows will close the groove. (i.e.: get a minimum of 3 liquid limit moisture contents).
- 10. Keep all moisture content samples in the oven for at least 16 hours.
 - 11. After the samples in the tares are completely dry, determine and record dry weights.
 - 12. Calculate the moisture contents (see moisture content procedures).
 - 13. Plot the liquid limit data points on the number of blows versus moisture content standard graph.
 - 14. Draw a best fit line through the points on the graph. The liquid limit is the moisture content where the line crosses 25 blows.
 - 15. Calculate the average moisture content for the plastic limit moisture samples.
 - 16. Subtract the plastic limit from the liquid limit to determine the plasticity index.

Test Method Title: Flexible Wall Permeability

GEL Author(s) of Procedure: John Tavassoli/James Stalcup

Approval By:

Reference No.: ASTM D 5084, D5887, EPA 9100, US COE EM-1110-1960

Test Category: Physical test method

Material Applicability: Soil, waste, and soil-like material

Target Property: Hydraulic conductivity/permeability

Units of Test Results: cm/s

Test Specimen Size: Minimum 1 in. (25 mm) long, 1 to 12 in. (25 to 305 mm) in diameter, typical specimen, 2 in. (51 mm) long and 2.8 in. (71 mm) in diameter

No. of Specimens Tested: 1

Test Equipment: Balance, calipers, de-aired water, filter paper, latex membranes, membrane stretcher, o-rings, permeability cell and panel, porous stones, pressure transducer, soil specimen trimming tools, split mold, balance

Test Equipment Calibration: See Laboratory Equipment Calibration and Maintenance Record binder

Test Speed: Material dependent

Deviations: Any deviations from the standard testing procedure shall be documented on the data sheet(s), and, if requested, reported in the final report submitted to the client.

Standard Operating Procedures:

1. Prepare a permeability test specimen using the following:
 - A. Shelby tube sample (undisturbed sample):
 1. remove the wax from the top of the material in the shelly tube;
 2. extrude material from tube moving the material in the same direction as it entered the tube; record any visible disturbances of the sample or irregularities (a sketch is often helpful);
 3. choose a representative portion of the sample and trim a test specimen to the desired height;

4. fill any voids along the sides with excess soil trimmed from the sample and smooth out vertical grooves;
5. square the ends of the specimen; and
6. record the specimen conditions (i.e., length, diameter, moist weight, and moisture content);

B. Remolded sample:

1. to form a standard permeability specimen with a height of 2.4 in. (60 mm) and a diameter of 2.8 in. (71 mm), prepare approximately 800 to 1000 g of a representative sample of the material to the target moisture content at least 16 hours prior to remolding; more time is recommended for soils/materials which are suspected of being fat clay; more material may be required for large/non-standard remolds;
2. determine the moisture content of the prepared soil using a minimum of 2 specimens;
3. define the length and diameter of the specimen to be remolded; standard permeability specimens are remolded in a split mold and are 2.4 in. (60 mm) long and 2.8 in. (71 mm) in diameter; the split mold has a length of 7.2 in. (183 mm) and a diameter of 2.8 in. (71 mm); the use of a stretched latex membrane within the split mold, used with less cohesive soils, may reduce the diameter of the specimen; the use of a plastic sleeve within the split mold, used to prevent soil from adhering to the sides of the mold, may reduce the diameter of the specimen;

Note: A target dry unit weight value and a moisture content value should be specified by the client for the sample remolding. Alternatively, the remold condition may be specified as a percentage of the maximum dry unit weight, determined by Proctor compaction, and a moisture content value referenced to the optimum moisture content. The actual remolded test specimen must be within ± 1.0 pcf of the target dry unit weight and ± 1.0 percent of the target moisture content. The goal in following these procedures is to form a uniform and homogeneous test specimen:

4. calculate the volume of the specimen to be remolded;
5. determine the required amount of dry material to be placed in the mold based on the target dry unit weight and the volume of the specimen;
6. calculate the required moist weight of material using the moisture content determined from the prepared soil (this may be slightly different than the target value);
7. dividing the calculated moist weight of material by the number of lifts to be used yields the weight of moist soil to be placed in each lift of the remolded specimen; usually 5 lifts are used for 2.4 in. (60 mm) long specimens;

8. place representative samples of the moist material for each lift in plastic bags;
9. take 2 representative moisture content specimens from the prepared material and label them as "actual";
10. the remolded specimen is compacted into the mold using the following: place the moist material for the first lift into the mold loosely and uniformly; using a hand tamper, approximately 9.4 in. (240 mm) long with a 1.5 in. (38 mm) diameter foot and a weight of 1 lb. (415 g), compact the soil to the required lift height; a consistent tamping energy should be used for each blow of the hand tamper; measurements from the top of the mold to the top of the soil should be periodically made until the soil reaches the correct height; each lift must be within ± 0.02 in (0.5 mm) of the target lift height; lightly scarify the surface of the lift; place additional lifts in a similar manner to the placement of the first lift; a tamping energy equal to that used to compact the first lift should be used for additional lifts (i.e., an equal number of blows with the tamper); and the final lift should not be scarified;
11. weigh the mold and remolded material prior to removal from the mold;
12. remove the specimen from the mold; observe and record the structure of the specimen;
13. square the ends of the specimen by trimming approximately 1 to 2 mm from each end;
14. record the length, diameter and moist weight; place the remolded specimen in a bag to prevent changes in moisture content; and
15. the specimen should be placed in a permeability cell within 1 hour of remolding unless otherwise specified.

C. Geosynthetic Clay Liner (GCL)

New ASTM Standard D 5887 for GCL **Method/Procedure being developed**

2. After preparing the specimen, place the specimen carefully in a permeability cell using the following configuration (top to bottom): top platen/porous stone/filter paper/test specimen/filter paper/porous stone/base platen.
3. Place one or preferably two latex membranes around the specimen and seal the ends with O-rings;
4. Assemble the cell and check for leaks using less than 5 psi Cell Pressure (CP); a leak check should not be performed when a very low consolidation pressure is specified.

5. Fill the cell and reservoirs with de-aired water; and bleed trapped air from the tubing and platens; slight Back Pressure (BP) may be used to help this process.
6. Check to insure that all valves are properly set up prior to starting saturation.
7. Start saturating the sample using the outflow reservoir, the panel will maintain an effective stress of 1.5 psi (Note: if an effective stress higher than 1.5 psi is specified during saturation the panel configuration must be changed) set the CP to 5 psi and increase by 5 psi every 1/2 hour until 45 psi is achieved. Higher CP and BP may be required to fully saturate some specimens.
8. After 12 to 24 hours of sustained CP and BP, check the "B" value using the pore pressure transducer; a minimum B value of 0.95 must be attained; consult the laboratory manager or project manager if the "B" value is less than 0.95.
9. Using the pressure transducer set the required CP and BP and consolidate the specimen in the following manner:
 - A. Record the BP and close the valves to the specimen;
 - B. Increase the CP to the required value such that the difference between the CP and BP is equal to the required consolidation pressure; and
 - C. Open the valves to the specimen and record the burette readings at 0, 0.1, 0.25, 0.5, 1.0, 2.0, 4.0 min. and periodically thereafter.
10. When primary consolidation is complete, apply a hydraulic head across the specimen and monitor the test with time using the following:
 - A. commonly, a falling head hydraulic gradient configuration is used to permeate the specimen (i.e., falling inflow and rising outflow); other configurations may be used;
 - B. record the inflow and outflow water level (burette) readings with time; reading intervals will vary from one specimen to another depending on material type and compaction;
 - C. readings should only be obtained if the change in burette height is greater than 0.2 in. (5 mm) and less than 4.0 in. (100 mm) for subsequent readings;
 - D. permeability (k) values should be determined at each reading by entering the time and burette readings into the programmed calculator on the panel; and
 - F. The test is considered stable and may be ended when the variation of k during 3 consecutive readings is less than 25 percent and a variation of less than 25 percent between the inflow and outflow volume have been achieved.
11. When the k value is stable and the inflow and outflow volumes are approximately equal the calculations should be checked using another calculator, initialed by the checker and marked so that it can be dismantled.

12. Dismount the specimen and record the final length, diameter, and moist weight; place the entire specimen in the oven to determine the final moisture content.
13. Report the average of the last 3 to 4 permeability values as the hydraulic conductivity in cm/s.

Prepared for:

Hercules Incorporated
Hercules Plaza
1313 North Market Street
Wilmington, Delaware 19894-0001

REVISION 0
HEALTH AND SAFETY PLAN

TERRY CREEK SITE
BRUNSWICK, GEORGIA

Prepared by:



GEOSYNTEC CONSULTANTS

1100 Lake Hearn Drive, NE, Suite 200
Atlanta, Georgia 30342

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1. INTRODUCTION

1.1 Terms of Reference

This Health and Safety Plan (HASP) was prepared for Hercules Incorporated (Hercules). The HASP was prepared by Mr. Leo F. Gentile, P.G., and it was reviewed by Mr. Charles A. Spiers, P.G., in accordance with the internal peer review policy of GeoSyntec.

1.2 Scope and Applicability of the Health and Safety Plan

This HASP describes measures that will be taken to ensure the protection of site workers during sampling at the Terry Creek Site (work site) near Brunswick in Glynn County, Georgia. The scope of work is described in detail in the document "*Sampling and Analysis Plan - Terry Creek Site, Brunswick, Georgia*" [GeoSyntec, 1997a]. On-site work includes, but is not limited to, collecting samples of soil, sediment, and dredge spoils for chemical analysis for toxaphene. The primary areas that will be investigated include a dredge spoil area, the marsh areas around the dredge spoil area, the berm surrounding the dredge spoil area, Dupree Creek, portions of Terry Creek, and the Outfall Ditch from the Hercules plant.

This HASP was prepared in accordance with the USEPA's Standard Operating Safety Guides [USEPA, 1992]. In addition, the plan complies with applicable Occupational Safety and Health Administration (OSHA) regulations and with hazardous waste operations and emergency response (HAZWOPER) requirements as specified in Title 29 Code of Federal Regulations, Part 1910 (29 CFR 1910).

1.3 Organization of the HASP

The remainder of this HASP is organized as described in the bullet items listed below.

- Section 2 describes the site, site history, and scope of work to be performed.
- Section 3 lists the key personnel who will manage and conduct the work at the site and identifies the personnel responsible for directing and administering the site health and safety program.
- Section 4 presents the hazard assessment and an analysis of the health and safety risks of the variety of tasks and operations to be performed in carrying out the required actions.
- Section 5 describes the medical surveillance requirements for on-site personnel.
- Section 6 describes personnel training requirements for on-site personnel.
- Section 7 defines the levels of protection, the types of personal protective equipment (PPE), and inspection and protective program reassessment requirements of the various tasks and operations.
- Section 8 describes site control measures including communications and definition of work zones.
- Section 9 describes the decontamination plan including definition of levels of decontamination for personal protection, equipment decontamination, and decontamination waste disposal.
- Section 10 discusses the frequency and types of air monitoring and sampling that will be performed during activities at the site.

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- Section 11 outlines the site health and safety standard operating procedures (SOPs), including availability of nearest medical assistance, safe working practices, and emergency alarm procedures.
- Section 12 presents the emergency response contingency plan.
- Section 13 describes confined space entry procedures.
- Section 14 presents a spill containment program for the work site.
- Section 15 describes the hazard communication and on-site record keeping and reporting program established to ensure that the presence and nature of known hazards on the work site will be communicated to all on-site personnel.
- Section 16 covers the site visitors policy.

2. SCOPE OF WORK

2.1 Scope of Work

The anticipated work tasks that will be performed are listed in the following bullet items.

- *Site Mobilization and Demobilization.* Equipment for site operations will be mobilized to the site prior to sampling activities. The equipment will be demobilized after work activities are finished.
- *Sampling.* Samples will be collected from the Terry Creek Site to provide additional information to characterize the nature and extent of toxaphene at the site. The samples to be collected include:
 - surface sediments in the marsh area;
 - sediment from Terry Creek, Dupree Creek, and the Hercules Outfall Ditch; and
 - soil and subsurface water samples from a Dredged Spoil Area.
- *Personnel and Equipment Decontamination.* All personnel and equipment, that comes into contact with contaminated materials will be decontaminated according to procedures outlined in this HASP and in the Sampling Plan.

3. PROJECT ORGANIZATION AND RESPONSIBILITIES RELATIVE TO HEALTH AND SAFETY

3.1 Overview

This section describes the job title and responsibilities of key health and safety project personnel. These personnel include facility representatives, project managers, site supervisors, subcontractors, and the site health and safety officer. Figure 2 is an organization chart showing key health and safety personnel for the removal action at the work site. Each key position is described below.

3.2 Hercules Incorporated

The Hercules project coordinator's responsibilities include oversight and technical input of the removal action sampling activities. The activities also include oversight of the analytical laboratory and validation of the analytical data. Mr. Timothy D. Hassett is the Project Coordinator for Hercules. Mr. Wayne Quinn will be the Site Field Engineer for Hercules.

3.3 Corporate Health and Safety Officer

The GeoSyntec Corporate Health and Safety Officer is responsible for developing and overseeing the corporate health and safety program. The GeoSyntec Corporate Health and Safety Officer will provide direction to the GeoSyntec Project Manager and/or the Project Health and Safety Officer, as necessary, on issues of health and safety.

3.4 GeoSyntec Project Manager

The project manager is responsible for the management of all aspects of a project including health and safety. Applicable health and safety tasks include the following: (i)

ensuring that all project personnel receive appropriate health and safety training before commencement of field activities; and (ii) ensuring that the necessary equipment and facilities are available to implement the health and safety plan.

The project manager may assign some tasks to task managers or to the site health and safety officer for implementation. The project manager is responsible for forwarding a copy of the HASP to all key health and safety personnel.

3.5 GeoSyntec Site Supervisor

The project manager may assign tasks within a project to one or more task managers or to the site supervisor. The site supervisor is responsible for the following: (i) ensuring that the health and safety aspects for their particular tasks are addressed; (ii) implementing appropriate work practices; and (iii) notifying the project manager of any changes in work conditions which affect the health and safety aspects of the task.

3.6 Subcontractors

It is not anticipated that subcontractors will be retained for this scope of work. In the event that they are needed, all subcontractors will be provided with a copy of this HASP. The HASP must be reviewed by all subcontractors. Furthermore, subcontractors are required to comply with all applicable and appropriate Federal, state, and local laws, standards, and regulations. Subcontractors may also be required to provide their own health and safety plan for work at the site. The subcontractor health and safety plan must be at least as stringent as this HASP.

3.7 Site Health and Safety Officer (SHSO)

The site health and safety officer (SHSO) is responsible for the implementation of the HASP, including all task-specific HASP addendum. The SHSO has the authority to

modify or stop any work on the site if there is an imminent danger to the health and safety of site workers or the general public.

The SHSO will also maintain files of medical and training certificates and will maintain a health and safety log. The health and safety log will be maintained in a bound field book. Each page of the book will be numbered, dated, and signed. The time and circumstances of each entry will be recorded. Health and safety comments entered in other field books will also be considered part of the health and safety log.

4. HAZARD ASSESSMENT

4.1 Introduction

The potential hazards that may be encountered during the sampling activities at the work site are variable. Of primary importance are the potential chemicals of concern and hazards associated with the work that will be performed. This hazard assessment is organized accordingly and presents information on the site contaminants and the potential hazards associated with various tasks. The chemicals of concern and exposure information are presented in Section 4.2. A task-by-task hazard analysis is presented in Section 4.3. Methods of control for these hazards are discussed in Section 4.4.

4.2 Chemicals of Concern

The chemical constituent of concern identified for the Terry Creek Site is limited to toxaphene. Toxaphene has been identified in dredge spoil and marsh sediments at concentrations up to 430 milligrams per kilogram (mg/kg). The chemical and physical chemical properties of toxaphene are summarized in Table 1.

4.2.1 Health Hazards

Health hazards, including potential routes of exposure; symptoms and effects of chemical exposure, target organs, and carcinogen classification associated with the principal constituent of concern are presented in Table 2. Occupational exposure limits, including OSHA's Permissible Exposure Limits (PELs), the American Conference of Governmental Industrial Hygienists' (ACGIH) Threshold Limit Values (TLVs) and the National Institute for Occupational Safety and Health's (NIOSH) Recommended Exposure Limits (RELs) are presented in Table 3. This information will be utilized in the task-by-task risk analysis in Section 4.3.

4.3 Task-by-Task Risk Analysis

4.3.1 Overview

A task-by-task health and safety risk analysis has been performed for the tasks associated with this project based on the chemicals of concern. This analysis is provided in matrix form in Table 4.

4.3.2 Task-by-Task Risk Analysis Matrix

Each task has been evaluated as to the chemical, physical, and biological hazards associated with the implementation of the task and the chemicals of concern associated with the site. If additional tasks are added to the project work plan, or additional hazards develop, each task will be reevaluated and the risk analysis matrix will be updated.

4.4 Hazard Discussion and Mitigative Controls

4.4.1 Overview

In this section, the hazards listed in the task-by-task risk analysis matrix are discussed and the controls to be implemented task-by-task to mitigate hazards are provided. These hazards may be controlled by the use of administrative actions (i.e., change in personnel, training, scheduling, etc.); engineering designs or procedures (i.e., use forced ventilation, etc.); or by the use of personal protective equipment.

4.4.2 Exposure to Toxic Atmospheres

The potential of exposure to toxic atmospheres may exist around any area that may be contaminated. By nature of the work to be performed, varying concentrations of

toxic airborne contaminants may be generated. In the disturbance of affected soils and dust, the human sense of smell is not sufficient to provide adequate warning of unsafe levels of airborne substances. Where affected materials exist or are suspected to exist, frequent air monitoring will be performed utilizing direct-reading instruments as presented in Section 10. Action levels, based on the occupational exposure limits are listed in Section 7.3.1. Appropriate action will be taken, depending on the air monitoring data, to protect workers as directed by the action levels.

Skin absorption is also considered a potential route of entry into the body if the skin is exposed to the constituent of concern. To protect workers from skin exposure, proper personal protective equipment (PPE) including protective gloves and suits will be utilized as described in Section 7. Workers can prevent skin contact by staying cognizant of their surroundings (i.e., try not to kneel or sit in contaminated areas; preventing slip, trip, and fall hazards; etc.).

4.4.3 Heat Stress

Heat stress is a significant hazard associated with this project. The danger of heat stress is aggravated by the factors listed below.

- The site is unshaded in many areas and is located on an inland tidal marsh on the Georgia coast. In the summer the climate is very hot and humid.
- Much of the ground is highly reflective of solar radiation. This is due to the water present in many areas. This condition increases the chance of sunburn and dehydration.
- Due to the requirements of this project, PPE (i.e., either Level B, C, or D) will be worn. PPE retards heat radiation and evaporation of moisture from the surface of the skin, which significantly impedes the body's natural means of cooling.

4.4.3.1 Types of Heat Stress

Heat stress is progressive in intensity and consists of three levels from mildest to most severe: (i) heat cramps; (ii) heat exhaustion; and (iii) heat stroke. Heat cramps cause mild to moderate discomfort and impede work performance. The symptoms of heat cramps are spasms in the abdomen and limbs. Frequent rest periods and fluid intake are appropriate measures to reduce and eliminate heat cramps.

Heat exhaustion causes sickness that may last for several hours to several days. Heat exhaustion results from dehydration. Symptoms of heat exhaustion include irritability, loss of judgment, pale, clammy skin, profuse sweating, headaches, nausea, pounding heart, and dizziness. If any of these symptoms occur, site workers should leave the site, decontaminate properly, find a shady location, drink fluids slowly, and if necessary, take a cool shower or bath. Due to the symptoms of irritability and loss of judgment, site workers must observe each other and may be required to force affected workers to take appropriate action.

Heat stroke is the most serious stage of heat stress and is life threatening. It occurs when the body temperature regulating system is no longer functioning properly. Heat stroke symptoms include hot, dry skin, a high fever (i.e., over about 106 degrees F), dizziness, nausea, rapid pulse, and unconsciousness. If heat stroke occurs, immediate action is required to lower the body temperature and secure medical attention. Application of ice packs, cold baths, and ice baths are appropriate first aid principles.

4.4.3.2 Heat Stress Safety Program

Heat stress will be monitored using the following program. The plan presented for monitoring and mitigating heat stress applies only to outdoor sampling and remediation activities during the hot season of the year.

- Workers will observe one another throughout the day for symptoms of heat stress.
- Medical-type ear thermometers will be used to monitor deep body temperature. Measurements will be taken on all workers at the start of work, scheduled rest periods, end of work and whenever a worker complains of or is observed to have symptoms of heat stress.
- Pulse will be measured and recorded whenever deep body temperature is measured and at the beginning of every rest period.

Heat stress will be mitigated using the following program.

- Work will be scheduled and paced to reduce the potential for heat stress. Under severe conditions, break time may exceed work time.
- A clean zone will be established in close proximity to each work site. The clean zone will contain decontamination facilities, a shaded area, chairs, and water (or electrolyte beverage). In this case it is assumed that the boat used for sampling site access will be designated as a clean zone.
- If a person's pulse rate exceeds 110 beats per minute at the beginning of the rest period, the work period will be shortened by one-third while keeping the rest period the same.
- If a person's deep body temperature exceeds 38°C (100.4°F), the person will be excused from work and placed on sick leave until his temperature recovers to normal.
- If a person exhibits symptoms of heatstroke, an ambulance will be called immediately and immediate action will be taken to lower the person's body temperature.

4.4.4 Trenching and Excavation Hazards

Trenching and excavation is not anticipated at the site. If sampling activities require trenching or excavating, entry into the trenches or excavations by site personnel will be kept to a minimum. If entry into a trench or excavation is necessary, OSHA trenching regulation found in 29 CFR 1226 Subpart P will be followed. Potential physical hazards associated with this work involve: (i) injury from heavy equipment; and (ii) collapse of the trench or excavation.

4.4.5 Equipment Hazards

Equipment to be used in the sampling activities could include hand augers, brush-clearing tools, and other small tools. Personnel at the site should be familiar with safe operating procedures for any equipment they use or supervise. Only properly trained personnel will operate such equipment.

Injury from the equipment can be minimized by observing the following: (i) ensuring that safety equipment is operational on the heavy equipment (i.e., safety belts and back-up signals); (ii) observing safe operational speeds; (iii) ensuring that all personnel in the vicinity of the heavy equipment are aware of the proposed activities of the machinery (i.e., activities involving heavy equipment should be discussed in the morning safety meetings); and (iv) ensuring that no heavy equipment operators use intoxicating substances while operating the equipment.

4.4.6 Facility Structures

Sampling activities could involve walking on structures such as docks, ladders, or stairs. Before any workers are to walk on these structures, visual inspection shall be

made to evaluate their safe use, and to identify the need to repair before their use. Routine inspections will be performed to identify weakened structures.

4.4.7 Back Strain

Some aspects of this work may involve the lifting of heavy objects and therefore the potential for back strain. Site workers should observe safe lifting procedures as follows:

- avoid lifting objects that are too heavy without help;
- lift with the back in a vertical position - use legs for strength;
- wear a back support, if necessary; and
- let machinery do lifting when practical.

It is up to the supervisor for each work group, contractor, or subcontractor to decide if back supports are necessary for that crew.

4.4.8 Biological Hazards

The work area for this site is located adjacent to a tidal marsh and woodland. Wildlife hazards associated with the area include alligators, snakes, ticks, spiders, scorpions, bees, and fire ants. Snake chaps are recommended for anyone working in heavy brush. Anyone bitten by a snake will be taken to the hospital for treatment as appropriate. Persons working in vegetated areas should routinely inspect themselves for ticks. Tick bites that swell or discolor should be treated by a physician. Nuisance insects are also present, and include mosquitoes, gnats, and biting flies. The use of insect repellent may be necessary at the site. Workers should scan the work areas for

poisonous plants such as poison ivy and poison oak. Thoroughly washing any exposed body parts will help protect against infection.

4.4.9 Slip, Trip, Fall Hazards

These hazards result from unlevel surfaces, slippery surfaces, and hard to see objects located across walking paths (i.e., rope, cords, tools) and are responsible for over 60 percent of work-related injuries. Good housekeeping procedures must be followed at all times to prevent such hazards.

4.4.10 Falling Objects

The potential for on site hazards from falling objects is considered low. It is considered good health and safety practice to don head protection despite this low risk. Therefore, hard hats, safety glasses, and steeled-toed footwear may be required for personnel during the execution of all operations and in all areas on site.

4.4.11 Confined Space Entry Hazards

Confined space entry is not anticipated for this scope of work, however, all suspected confined spaces will be evaluated as per 29 CFR 1910.146. If entry into a confined space is required, the workers must understand and implement the confined space entry procedures as explained in Section 13.

4.4.12 Boat Hazards

Boat hazards will be potentially present whenever sampling or inspections are required over open bodies of water. Explosion, slip, trip, and fall hazards are always present with boating operations and one addressed elsewhere in this plan. In addition to those hazards, other items that must be addressed include the following:

Initial Safety Check (prior to boat launch)

- drain plug (where fitted) are in place;
- each person has a coast guard-approved personal flotation device (PFD); and
- safety equipment includes at least one coast guard-approved throwable PFD, one emergency signal (e.g., horn or air horn), and rope.

During launch

- launch boat from a safe area, avoid soft ground and slippery areas (to the extent possible, use a purpose-built ramp);
- secure and evenly distribute all loads; and
- avoid strong currents.

During operation

- avoid unnecessary movement, standing, etc.; and
- when possible, secure boat to a fixed station during sampling or inspection activities.

5. MEDICAL SURVEILLANCE REQUIREMENTS

5.1 Overview

All personnel who engage in the work involving potential exposure to hazardous substances for this project will be participants in a medical monitoring program for personnel involved in hazardous waste operations. This medical monitoring program must meet the minimum requirements of 29 CFR 1910.120(f). Certificates or letters (or copies thereof) documenting that each person is in a medical monitoring program and that each person is medically fit to work on hazardous waste sites will be maintained on site by the site supervisor and will be available for inspection at any time.

This section of the HASP discusses the general requirements for medical monitoring required in OSHA regulations for hazardous waste sites (i.e., 29 CFR 1920.120(f)).

5.2 Baseline or Preassignment Monitoring

The baseline medical exam will include:

- history and physical;
- full vision screen;
- blood chemistry and heavy metal screening;
- urinalysis;
- electrocardiogram (EKG);
- spirometry;

- chest X-ray (2 views);
- audiometry;
- hemocult slides (if recommended by the physician);
- tuberculosis (TB) skin test and tetanus toxoid; and
- flexible sigmoid (if needed).

5.3 Periodic Monitoring

A periodic exam will be required if any worker develops signs or symptoms related to the possible overexposure to hazardous substances or is exposed while unprotected in an emergency situation. Hence, should any worker believe that an exposure has occurred, the SHSO must be advised immediately. The scope of the periodic monitoring exam will be left to the discretion of the examining physician.

5.4 Exit Physical

The exit physical for a worker will be conducted upon termination of employment at the site. This physical will include all items listed for the baseline medical exam.

6. PERSONAL TRAINING REQUIREMENTS

6.1 Overview

This section provides an overview of the training background required for each person involved in on-site work during this project. Training related meetings are also discussed.

6.2 Preassignment and Annual Refresher Training

All project personnel who engage in the field work or decommissioning will have completed 40 hours of health and safety training for hazardous waste operations with annual eight hour refresher training as required under 29 CFR 1910.120(e). Certificates (or copies thereof) documenting this training will be maintained on site by the site supervisor and will be available for inspection at any time.

6.3 Site Supervisors Training

The site supervisor will have completed 40 hours of health and safety training for hazardous waste operations with annual eight hour refresher training as required under 29 CFR 1910.120(e). In addition, the site supervisor must also have completed eight hours of additional supervisory training. Certificates (or copies thereof) documenting this training will be maintained on site by the site supervisor and will be available for inspection at any time.

6.4 Training and Briefing Topics

Before any worker new to the project begins work on the site, that worker will read this HASP. Questions about the HASP will be answered by the SHSO. Alternatively, the contents of the HASP may be explained to the worker. When the worker and the SHSO are confident that the worker adequately understands this HASP, the worker will sign and date the affidavit in the front of this plan. Once a worker has signed the HASP, presented current certificates of training in accordance with 29 CFR 1910.120(e), and presented a current medical monitoring certificate and certification of respirator fit test, they will be issued a gate pass and will be permitted to work on-site. Respirator fit testing may be performed at the site.

Each morning before work begins, the SHSO or a designated representative will meet with all personnel and any subcontractors under his direction and review relevant health and safety issues. The morning safety meetings will focus on the following items:

- the nature of the hazards associated with the work to be performed that day (i.e. the hazard analysis);
- the procedures that will be used to mitigate the hazards;
- the emergency response plan;
- standard operating procedures; and
- brief refreshers of 29 CFR 1910.120.

The morning safety meeting, including the items discussed, will be documented in the site log book.

7. PERSONAL PROTECTIVE EQUIPMENT

7.1 Overview

This section of the HASP describes personal protective equipment (PPE) required to be worn by facility personnel. All equipment for each level of protection is mandatory except snake chaps and hearing protection. Snake chaps may be worn by personnel working outside in overgrown or brushy areas. Hearing protection may be required if the action levels for noise described in Section 7.3.3 are exceeded. The proposed definitions of levels of protection are presented in Section 7.2.

7.2 Levels of Protection

7.2.1 Overview

This section of the HASP describes the various levels of personal protection that will be worn at the site. The levels of protection will vary according to the proposed work task that will be performed on the site. Furthermore, the levels of protection may be modified if appropriate site monitoring indicates that the potential for exposure is either less or greater than expected. Levels C and D personal protective equipment are described in the following sections. Most of the site work is to be performed in either Level D or Level C.

7.2.2 Level C Personal Protective Equipment

Level C will be selected when the concentration and type of airborne hazardous substances is known and the criteria for selection of air-purifying respirators are met. The minimum equipment requirements for Level C are as follows:

- full-face air purifying respirator with appropriate cartridges;
- disposable coveralls: all joints must be taped;
- gloves: outer chemical-resistant (neoprene);
- gloves: inner, chemical-resistant (nitrile or equivalent);
- boots: chemical-resistant, steel toe (polyvinyl chloride (PVC), polyurethane, or equivalent);
- snake chaps (if required);
- hearing protection (if required); and
- hard hat.

If working in areas where contact with potentially contaminated liquids is possible, the disposable coveralls shall consist of Saranex coated Tyvek or equivalent, as approved by the SHSO.

7.2.3 Modified Level D Personal Protective Equipment

Modified Level D is to be selected when no respiratory hazards, but general dusty or dirty conditions exist. The minimum equipment requirements for Modified Level D are as follows:

- disposable coveralls ;
- gloves outer, chemical resistant (neoprene);
- gloves inner, chemical resistant (nitrile or equivalent);
- boots: chemical-resistant, steel toe (polyvinyl chloride (PVC), polyurethane, or equivalent);
- snake chaps (if required);
- hearing protection (if required);
- hard hat; and
- safety glasses with side shields.

7.2.4 Level D Personal Protective Equipment

Level D PPE is only appropriate when no respiratory or dermal protection is required. The minimum requirements for Level D are as follows:

- pants and shirt or coveralls;
- boots, steel toe or hip waders where appropriate;

- hard hat; and
- safety glasses with side shields.

7.3 Reassessment of Protective Program

The level of personal protection will be subject to change pending the results of on-site air monitoring (see section 10). However, in no case will PPE be reduced when unknown conditions exist at the site. Procedures for monitoring airborne hazardous substances (i.e., organics and heavy metals) are described in Section 10.

7.3.1 Action Levels for Airborne Constituents

Toxaphene on soil or sediment is not a volatile chemical of concern, therefore, the inhalation pathway is through airborne particles. The total particulate concentration in air that would have to be reached to exceed the threshold limit value-time weighted average (TLV-TWA) for toxaphene has been calculated using: (i) a maximum soil concentration of 430 mg/kg; and (ii) a TLV-TWA of 0.5 mg/m³. Based on this calculation, the maximum particulate concentration is 1,163 mg/m³. Because this value exceeds the 10 mg/m³ ACGIH LV-TWA for Particulates Not Otherwise Classified (PNOC), the action level for airborne constituents will be the PNOC TLV-TWA. Visual and instrumental monitoring will be performed during sampling activities to assure that particulate levels in the work area do not exceed this value. As noted in Section 10, Air Monitoring is only required when the generation of dust is possible.

7.4 Work Mission Duration

During activities that require Level C PPE, all personnel will, at a minimum, take breaks according to the following schedule:

- 15 minute break mid-morning;
- 30 minute break at lunch; and
- 15 minute break mid-afternoon.

More frequent breaks will be recommended if any workers show symptoms of heat stress (Section 4.3.3).

7.5 Chemical Resistance and Integrity of Protective Material

It is not expected that long-term contact with concentrated hazardous substances will occur during most aspects of this project. Therefore, protective equipment should primarily serve to protect against occasional contact and splashes of low concentrations of chemicals.

7.6 Inspection

The SHSO, or designate, will routinely inspect all PPE and monitoring equipment to ensure that it is in good operating condition. Monitoring equipment will be tested and calibrated daily. Any concerns regarding the condition of PPE or monitoring equipment will be evaluated immediately by the SHSO or designate.

8. SITE CONTROL MEASURES

The work site is a marsh area and access to the site is uncontrolled. Access to all sampling locations in the marsh and creeks will be by boat. Access to the dredge spoil areas will be by boat and then on foot. Therefore, there are no site control measures to be implemented.

8.1 Support Zone

The support zone consists of all areas of the work site that are not part of the areas to be sampled. The following activities will take place in the support zone: (i) automobile parking; (ii) supply and equipment storage; (iii) rest breaks; (iv) training meetings; and (v) observation of activities by personnel who do not meet the requirements for entering the sampling areas.

8.2 Exclusion Zone

An exclusion zone will be established around the active work area while activities occur within the area. This zone will extend beyond the work area a sufficient distance so that personnel and equipment may operate effectively without having to leave the exclusion zone. All entry into and exit from the exclusion zone will be through the CRZ.

Entry into the exclusion zone is limited to those personnel who meet the requirements listed below:

- the medical monitoring and training requirements established in Sections 5 and 6 of this HASCP are met;
- the appropriate level of personal protective equipment as established in Section 7 of this HASCP is worn;
- the need to be in the exclusion zone; and

- the adherence to decontamination procedures established in Section 9 of this HASP.

8.3 Contamination Reduction Zone

A CRZ will be established in an area that is contiguous with the exclusion zone. The CRZ will consist of the decontamination area as needed. The CRZ will be established daily, along with the exclusion zone, and will be based upon the planned activity. When the daily activities involve multiple and non-contiguous locations, an all-encompassing CRZ may be established to minimize the need for decontamination between work areas. All entrance into and exit from the exclusion zone will be through the CRZ. The CRZ is limited to personnel who are authorized to enter the exclusion zone.

9. DECONTAMINATION PLAN

9.1 Overview

Decontamination of personnel and equipment will take place during site sampling activities to prevent the spread of contamination into other areas, and to reduce exposure to other personnel and the environment. This section of the HASP presents the decontamination procedures to be used throughout the duration of the project.

9.2 Standard Operating Decontamination Procedures

9.2.1 Introduction

Hazardous substances must be removed from all personnel and equipment before leaving the exclusion zone. The procedures for performing decontamination are described in this section. The following procedures are discussed in this section: (i) personal decontamination; (ii) equipment decontamination; and (iii) disposal of decontamination wastes.

9.2.2 Personal Decontamination

A decontamination station will be established at the Hercules Boat Dock. Other stations may be established if deemed appropriate by the Field Manager. All equipment will be cleaned in the same phase. Personal decontamination will consist of the following procedures:

- Step 1: fill containers with wash and rinse solutions;
- Step 2: wash boots and gloves;
- Step 3: rinse boots and gloves;

- Step 4: remove tape if worn and dispose;
- Step 5: remove boot covers if used and dispose;
- Step 6: remove outer gloves;
- Step 7: remove disposable coveralls by rolling down and dispose;
- Step 8: wash and rinse inner gloves;
- Step 9: remove eye and/or respiratory protection;
- Step 10: wash respirators in disinfectant solution and dispose of expired cartridges;
- Step 11: remove inner gloves;
- Step 12: allow respirator to air dry;
- Step 13: wash the face, hands, and other exposed body areas; and
- Step 14: wash the hard hat or wipe with a wet towel.

9.2.3 Equipment Decontamination

All sampling equipment will be placed in plastic bags before it is removed from the exclusion zone. Sampling equipment will be cleaned in an area designated by Hercules site personnel. Monitoring equipment will be cleaned at the decontamination station described in Section 9.2.2. Monitoring equipment will generally be washed with water

and detergent. More specialized decontamination procedures may need to be used in specific cases, and will be evaluated on a case-by-case basis.

9.2.4 Disposal of Decontamination Wastes

Disposal of wastewater and solids generated during decontamination of sampling equipment will include containerizing this material in plastic buckets with lids. The material in these buckets will be consolidated in a drum. The materials will be handled according to State and Federal guidance.

10. FREQUENCY AND TYPES OF AIR MONITORING/SAMPLING

10.1 Overview

Air monitoring will be performed, as dictated by the known or suspected chemicals of concern for the work area. As discussed in Section 7.3, the action level for airborne constituents will be the PNOC TLV-TWA. Therefore, air monitoring will be limited to dust monitoring. Many of the samples collected will be saturated. Therefore, dust monitoring will be limited to areas or conditions where the generation of dust is possible.

Monitoring will be conducted in the breathing space of the workers. Air monitoring data will be entered in the site health and safety log book. This will include calibration data and all readings collected. Direct-reading measurements will be used to evaluate local and immediate hazardous situations. Air sampling will be used to characterize the specific constituents present and to predict which constituents might pose a hazard for future operations at the site, specifically the removal action.

10.2 Direct-Reading Monitoring Instruments

10.2.1 Dust Monitoring

A dust monitor, Mini-Ram Model PDM (or equivalent), will be used to identify the level of total dust particulates on a real-time basis for those activities where dust will be generated (i.e., during excavation activities). Action levels for dust are as follows:

- 0 to 10 mg/m³ above background in the breathing zone: Level D (if no other constituents are anticipated); and
- greater than 10 mg/m³: Level C (if no other constituents are anticipated).

Instructions for the dust monitor will be on file with the site supervisor. Calibrations are to be recorded in the site logbook.

10.2.2 Dust Suppression

Engineering controls will be implemented to minimize dust levels. Dust control (i.e., spraying water) will be implemented as deemed necessary by the SHSO. These controls are anticipated to consist of using hand pump sprayers to control dust in the immediate work area.

11. SITE HEALTH AND SAFETY STANDARD OPERATING PROCEDURES (SOPs)

11.1 Overview

This section gives details on standard safety guidelines to follow during all work tasks.

11.2 Buddy System

When participating in work activities at the Terry Creek Site, workers will use the buddy system. Each worker will be observed by one or more other workers for signs of problems. Buddies should enter and exit at the same time, stay close together and must maintain visual contact. Responsibilities of workers utilizing the buddy system include:

- providing assistance to the buddy;
- observing the buddy for signs of chemical or heat exposure;
- periodically checking the indicator on the buddy's respirator cartridge;
- periodically checking the integrity of the buddy's PPE; and
- notifying site personnel if emergency assistance is needed.

11.3 Site Communications Plan

11.3.1 Internal Communication

The ability to communicate hazards to site personnel and the SHSO will be available at each active work site. This will include a two-way radio and/or cellular telephone for contact with the SHSO or designated representative. Radios and/or telephones should be fully charged each night and tested each morning. Radios will remain on the frequency designated by the SHSO at all times.

Emergencies at the facility are signaled by five short blasts on the plant air horn or air whistle, depending on the location of the emergency. The all-clear signal is two long blasts. These signals will be preserved for all emergencies. Each active work site will have an air horn available.

11.3.2 External Communication

A cellular telephone will be the means of off-site communication. Any emergency or need of assistance by off-site personnel will be handled with this telephone. Daily inspection that the telephone is in good working order and periodic contact with off-site supervisors is essential.

11.4 Nearest Medical Assistance

The closest hospital to the site is the Southeast Georgia Regional Medical Center. This hospital is located approximately 1 mi (1.6 km) from the site on the corner of Parkwood and Kimble streets. From the Hercules plant take Riverside Drive north to Parkwood Drive. Turn left (west) on Parkwood Drive, and cross Glynn Avenue. The hospital is 0.6 miles west on the right. Figure 3 shows the route to the hospital. This figure will be posted at various locations around the site.

12. EMERGENCY RESPONSE/CONTINGENCY PLAN

12.1 Overview

In addition to the standard work practices, the emergency response and contingency plan applies to all personnel at all times and for all sites throughout the life of the project. This section outlines: (i) pre-emergency planning; (ii) personnel roles; (iii) evacuation and emergency procedures; and (iv) emergency equipment and facilities.

12.2 Pre-Emergency Planning

Medical emergencies due to injury or illness of project personnel are possible in association with this project.

12.2.1 Medical Emergencies

The site supervisor will prepare for medical emergencies before beginning work on the site by doing the items listed below:

- driving to the nearest hospital from the work site verifying the travel route;
- locating and testing the nearest telephone to the work site;
- ensuring that there is an adequately supplied first-aid kit available;
- ensuring that there is an adequate supply of cool, potable water in order to be used in the prevention and treatment of heat injury;

- ensuring that there are adequate facilities for washing-off hazardous wastes that may come in contact with personnel, which includes ensuring that there is an adequate supply of potable water to be used for washing;
- ensuring that there are adequate fire extinguishers available and that those extinguishers are fully charged;
- performing a reconnaissance of evacuation routes from the work site in the event of a general, facility-wide release of a hazardous substance; and
- ensuring that all key personnel on the site are adequately trained.

Portable safety showers will and eye wash stations will be located close to the work area. It is the responsibility of the SHSO to check that each station is in working order, or properly identify non-working stations and provide alternative facilities (if necessary).

12.2.2 Standard Safety Equipment

The site supervisor will ensure that the following emergency response equipment is available at each active work site:

- a first-aid kit;
- a fire extinguisher for the boat of the ABC type with at least a 20-pound rating;
- a properly filled and prepared eyewash device; and
- a cooler of ice water with an adequate supply of paper cups.

12.3 Personnel Roles and Lines of Authority

On-site supervisors for specific tasks must coordinate safety procedures with the SHSO. Workers should address safety concerns and report safety-related issues to their site supervisor, who will then report to the SHSO. Of course, ultimately, the health and safety for project personnel lies with the individual.

12.4 Evacuation Routes/Procedures

The potential for the need for emergency evacuation from the work site is considered to be very low. In the event that an emergency evacuation is required, personnel will be transported using the sampling boat to the Hercules dock. From that point, transport to medical attention will be either by ambulance or personal vehicle depending on the severity of the medical condition.

In the event of a severe medical emergency such as heatstroke, the cellular phone will be used to contact Hercules, to summon an ambulance, and to notify the medical center of the situation. The affected individual will be transported by boat to the Hercules dock where it will be met by the ambulance. The boat will follow the shortest navigable route to the dock. In the event that low tide conditions prevent the boat from reaching the dock, other evacuation means, such as air evacuation by helicopter, will be used.

12.5 Emergency Contact/Notification System

In the event of an emergency, the site supervisor or their designee is required to notify the following officials as soon as is reasonably possible:

- the SHSO;

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- the facility representative;
- the project manager; and
- the project coordinator.

12.6 Emergency Medical Treatment Procedures

In the event of a medical emergency, personnel will call the 911 emergency hotline in Brunswick and summon an ambulance. The emergency hospital for the Brunswick area, and the closest hospital to the site, is the Southeast Georgia Regional Medical Center. This hospital is located approximately 1 mi (1.6 km) from the site on the corner of Parkwood and Kimble streets. Before work on the site begins, the site supervisor should reconnoiter the route to the hospital.

12.7 Fire or Explosion

If possible without placing site workers in jeopardy, small fires may be extinguished utilizing fire extinguishers. Site workers should not attempt to extinguish fires that are too large or out of control. The local fire department will be called for assistance if the site workers cannot extinguish the fire. Notification will include any other hazards that the fire department should be aware of when they respond.

Following an explosion, first-aid should be administered to injured personnel provided that action does not endanger other personnel. If a fire results from the explosion, fire-fighting procedures should be the same as described above.

12.8 Emergency Equipment/Facilities

In the case of an emergency, first aid and fire extinguishers will be available at each work site. There will also be a method of communicating information to the SHSO. Any other equipment and facilities will be provided by municipal emergency response teams.

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13. CONFINED SPACE ENTRY

As discussed previously, confined space entry is not expected for this scope of work.

14. SPILL CONTAINMENT PROGRAM

As no liquid hazardous materials in any quantity are anticipated to be found during sampling activities, no special spill containment program is deemed necessary. If any liquid such as decontamination fluids are spilled, the site manager should provide enough commercially available absorbent material to recover the material. The area of transfer of the waste to its final container at the site should be covered in plastic.

15. HAZARD COMMUNICATION

The SHSO will establish a hazard communication program to ensure that site workers are fully informed of all known hazardous chemicals on the site. The hazard communication program will comply with the OSHA requirements in 29 CFR 1910.1200. The program will include a list of all known hazardous substances on site as well as Material Safety Data Sheets (MSDSs), or the equivalent, for each chemical. MSDSs for chemicals thought to be present at the site are available from the SHSO. The SHSO must also assure that each worker understands the hazards.

16. VISITORS

No visitors will be permitted at the sampling site work area without first receiving a site orientation from either the SHSO or Hercules field engineer. The site orientation will include the following: (i) description of site hazards; (ii) description of current site activities; and (iii) general rules and SOP's.

No visitor to the work site will be permitted beyond the Support Zone of the work site (i.e., they will not be permitted to enter the areas to be sampled, unless they have first met: (i) the mandatory health and safety training requirements for personnel set forth in Section 6 of this HASP; (ii) the medical surveillance requirements described in Section 5 of this HASP; and (iii) respirator fit-test certificate. Also, they must utilize all personal protective equipment (PPE) (see Section 7) specified in this HASP for the particular to be visited.

TABLE 1

**CHEMICAL AND PHYSICAL PROPERTIES OF
CONSTITUENTS OF CONCERN**

Contaminant	Flash Point (°C)	Upper Explosive Limit	Lower Explosive Limit	IDLH (mg/m³)	Ionization Potential (eV)	Density (g/cm³)
Toxaphene	34.4 and 135 (closed cup)	NA	NA	200	NA	1.65 at 25°C

TABLE 2
HEALTH HAZARDS

Contaminant	Physical Description	Routes of Exposure	Symptoms & Effects	Target Organs	Carcinogenes Classification
Toxaphene	Amber waxy solid, pleasant odor	Skin contact Dust inhalation Dust ingestion	Nausea; vomiting salivation, muscle spasms skin irritation	Eyes Skin Central Nervous System Liver Kidneys	OSHA - No EPA - B2 NTP - 2B IARC - Group 2B ACGIH - A3 NIOSH - yes

TABLE 3
OCCUPATIONAL EXPOSURE LIMITS

Chemical Constituent	ACGIH[*] 8-Hour TWA TLV (mg/m³)	OSHA^{**} 8-Hour TWA PEL (mg/m³)	NIOSH^{***} 10-Hour TWA REL (mg/m³)
Toxaphene	0.5	0.5	ALARA

C Ceiling Limit

* ACGIH Threshold Limit Values, 1995-1996.

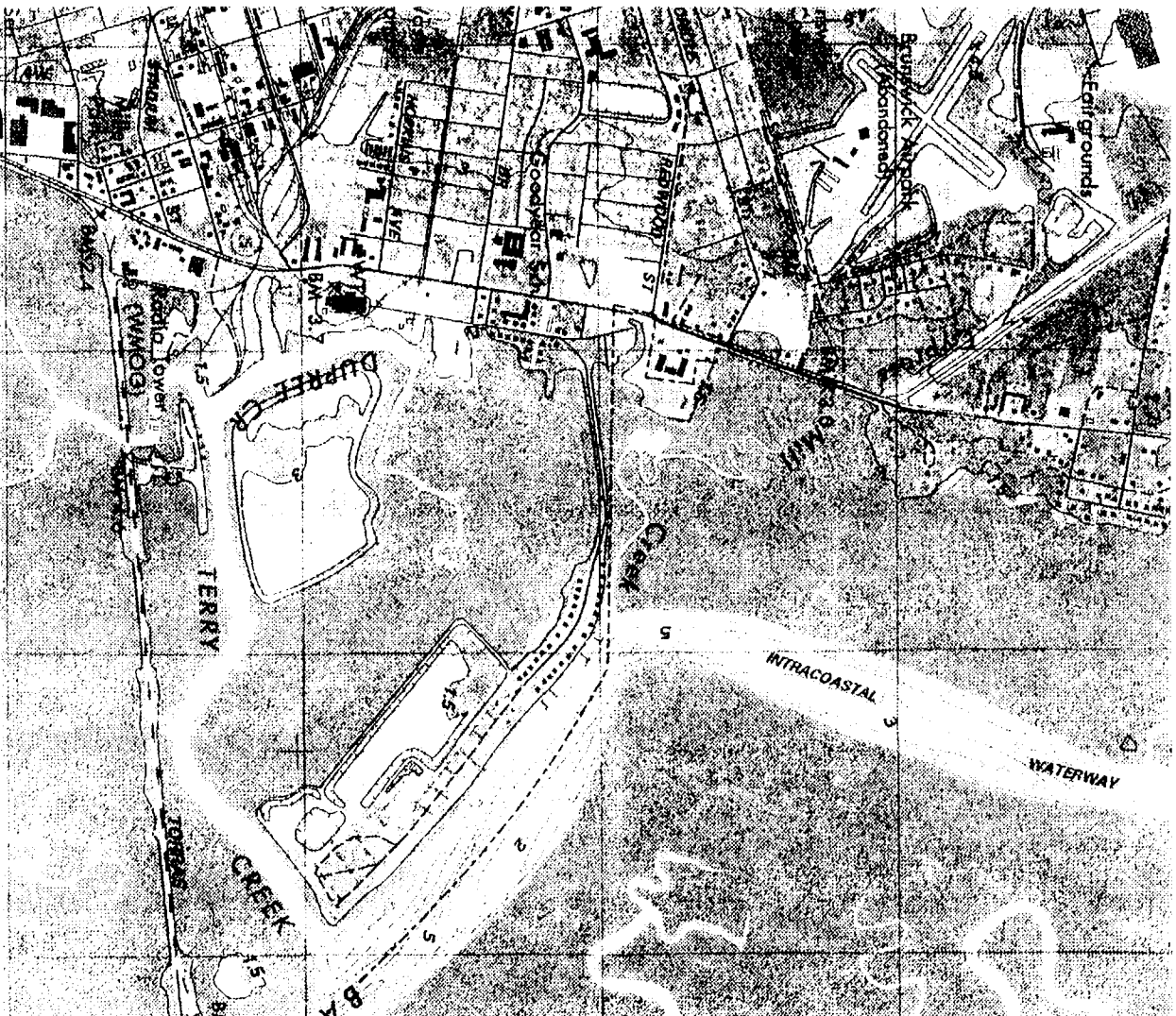
** OSHA Permissible Exposure Limits, 29 CFR 1910.1000.

*** NIOSH Recommended Exposure Limits, 1994.

mg/m³ = milligram per cubic meter

ALARA = as low as reasonably achievable.

LOCATION OF THE TERRY GREENS SITE BROWNSWICK, GEORGIA 2 4 U.S.



SOURCE: USGS BROWNSWICK EAST
7.5 MIN TOPO QUADRANGLE (1982)

1000 2000 3000 Feet

FIGURE NO.

PROJECT NO. G00270

DOCUMENT NO. GA970905

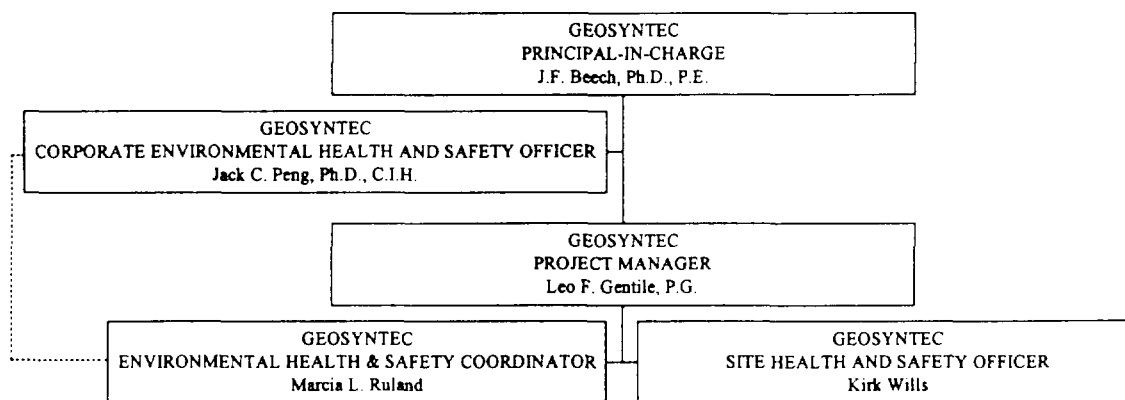
FILE NO. FIG. APR



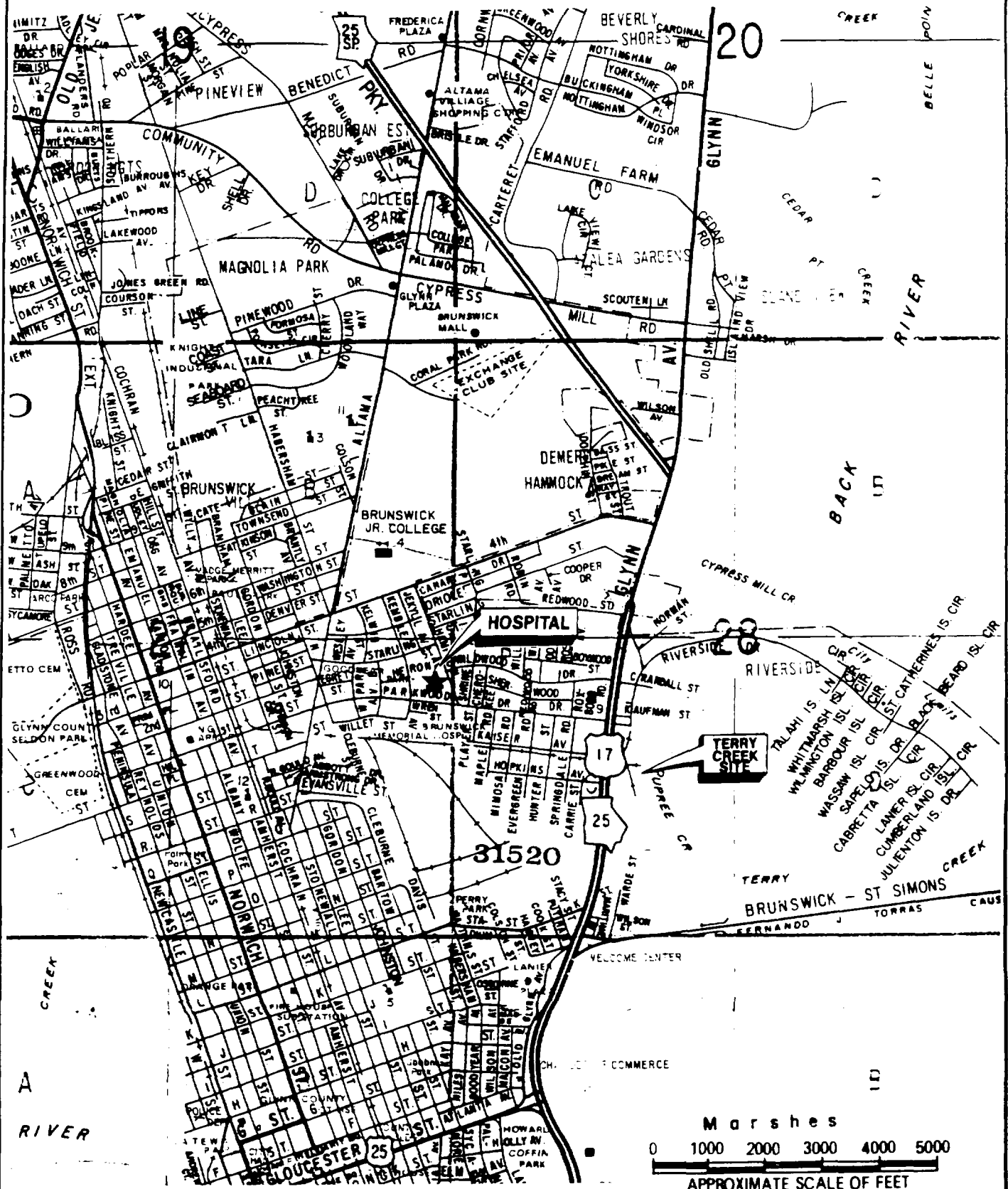
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ATLANTA, GEORGIA

FIGURE 2
KEY HEALTH AND SAFETY PERSONNEL
TERRY CREEK SITE
BRUNSWICK, GEORGIA



HOSPITAL LOCATION PLAN



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FIGURE NO.	3
PROJECT NO.	GQ0270-02
DOCUMENT NO.	GA970905
FILE NO.	DF



GEOSYNTEC CONSULTANTS

1100 Lake Hearn Drive • Suite 200
Atlanta, Georgia 30342-1523 • USA
Tel. (404) 705-9500 • Fax (404) 705-9400

22 August 1997

Mr. Leo Francendese
U.S. Environmental Protection Agency
Region IV WMB/ERRB
Atlanta Federal Center
100 Alabama Street
Atlanta, Georgia 30303

Subject: Submittal of Figures
Terry Creek Site
Brunswick, Georgia

Dear Leo:

Enclosed are three copies of the figures of the additional sampling areas at the Terry Creek Site.

Please let us know if we can provide you with any additional information regarding this project.

Sincerely,

Charles A. Spiers, P.G.

Attachments

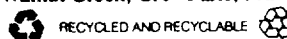
-/LEO.LTR

Corporate Office:

621 N.W. 53rd Street • Suite 650
Boca Raton, Florida 33487 • USA
Tel. (561) 995-0900 • Fax (561) 995-0925

Regional Offices:

Atlanta, GA • Boca Raton, FL • Chicago, IL
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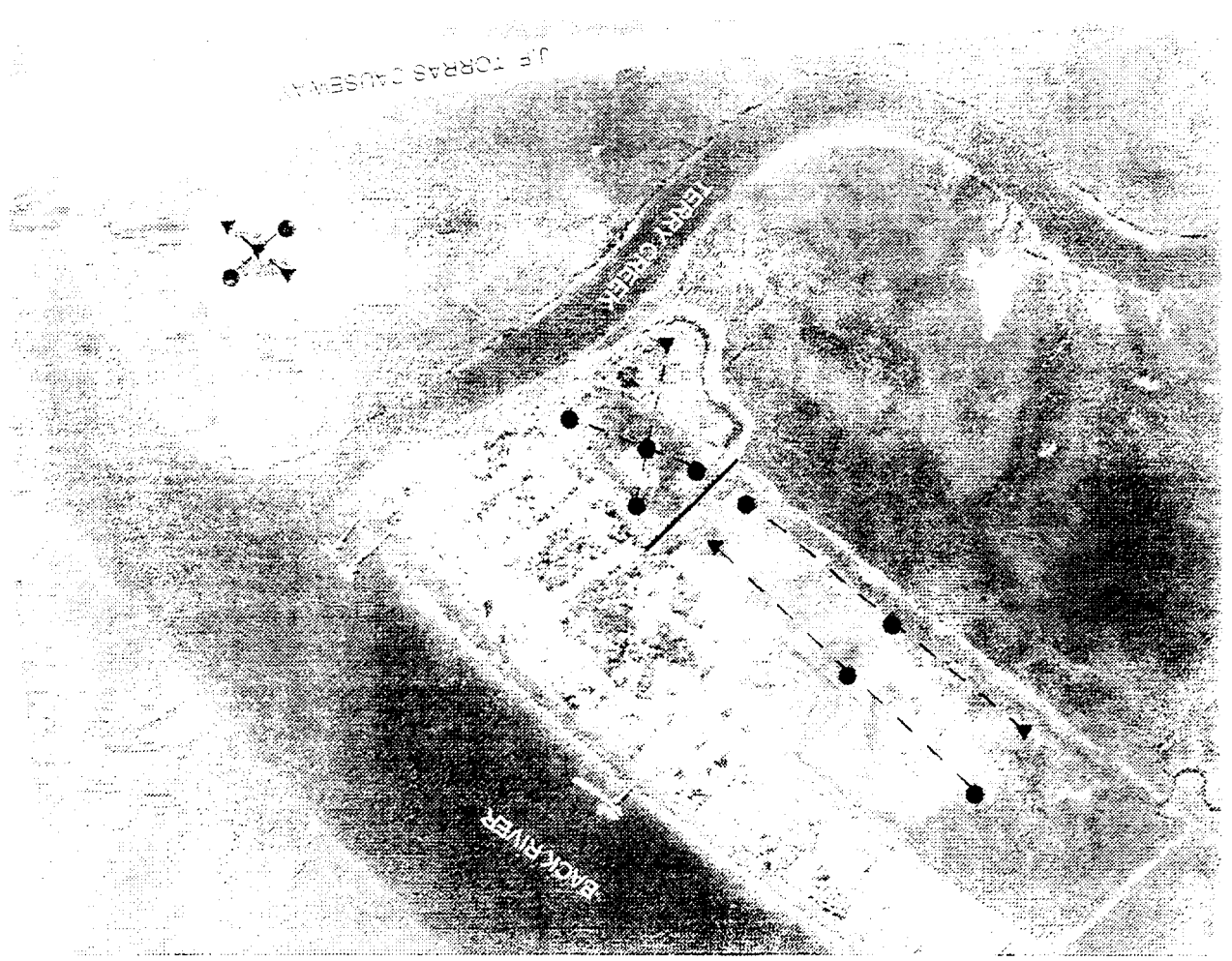


Laboratories:

Atlanta, GA
Boca Raton, FL
Huntington Beach, CA

500 0 500 500 500 Feet

LEGEND
 (Symbol descriptions)



ADDITIONAL SAMPLING LOCATIONS